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Measurements of High-Temperature High-Pressure Processess: A Summary Report

Acurex Corp.
Mountain View, CA

Prepared for

Industrial Environmental Research Lab.
Research Triangle Park, NC

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April 1982

MEASUREMENTS OF HIGH-TEMPERATURE HIGH-PRESSURE PROCESSES -- A SUMMARY REPORT

by

L. Cooper, M. Shackleton
Acurex Corporation
Energy & Environmental Division
485 Clyde Avenue
Mountain View, California 94042

Contract 68-02-2153

Project Officer

W. Kuykendal

Prepared for

U.S. Environmental Protection Agency
Research Triangle Park, North Carolina 27711

INDUSTRIAL ENVIRONMENTAL RESEARCH LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
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16. ABSTRACT

The report summarizes an assessment and development of technology required to perform high-temperature high-pressure (HTHP) particulate sampling. In addition to efforts devoted to devloping and testing an HTHP sampler for the EPA/Exxon Miniplant, experience was gained in the design aspects of HTHP sampling equipment and testing procedures. This experience is highlighted in the report. A background study and planning effort was directed toward possible future sampling efforts in a coal gasification plant. A state-of-the-art review of HTHP sampling was also performed. To document the materials collected, a bibliography of articles, reports, and books relating to HTHP sampling was compiled. A list of persons interested in this technology is also included. Results demonstrated that particle size distribution and samples suitable for chemical analysis can be obtained with the apparatus described in the report.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS		b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group	
Pollution	Fluidized Bed Processing	Pollution Control	13B	
Sampling		Stationary Sources	14B	07A
Temperature	Combustion	Particulate Sampling	14G	21B
Pressure		High Temperature		
Dust		High Pressure	11G	
Coal Gasification		Extractive Sampling	11H	

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FOREWORD

New energy conversion processes are being developed to utilize coal. The best time to examine these processes for potential damage to the environment is during their development cycle. The Industrial Environmental Research Laboratory is engaged in work which examines pollution potential for emerging coal conversion technologies.

Several of these emerging technologies, for example pressurized fluidized bed combustion and gasification processes, involve gas streams at high temperature and pressures which contain particulates. Since these particulates generally must be removed from the gas stream, hot gas cleaning devices are also under development. In order to evaluate the effectiveness of these hot gas cleaning devices, particle measurement and sampling equipment is needed which can operate in these severe environments. This report examines some of the problems associated with particle sampling in high temperature and pressure environments.

ABSTRACT

The major focus of this program under EPA contract 68-02-2153 was that of particulate sampling in advanced coal conversion technologies. Work performed was to assess and develop the technology required to perform high-temperature high-pressure particulate sampling. In addition to the effort denoted to development and testing of an HTHP sampler for the EPA/Exxon Miniplant, experience was gained in design aspects of HTHP sampling equipment and testing procedures and is highlighted in this report. A background study and planning effort was directed toward possible future sampling efforts in a coal gasification facility. A state-of-the-art review of HTHP sampling was also performed. As a means of documenting the materials collected, a bibliography of articles, reports and books relating to HTHP sampling was compiled. Further a mailing list of persons interested in this technology is included in the report.

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SECTION 1

EXECUTIVE SUMMARY

1.1 AN OVERVIEW OF THE PROBLEM

Sampling for particulate matter in hostile environments is a technology that is still in the early stages of development. The need for these techniques, however, is yet to be fully realized. High-temperature or high-pressure (HTHP) sampling is integral to the resolution of environmental concerns resulting from the development of immerging energy conversion technologies. There are also potential applications for HTHP sampling in current technologies, including those in the petrochemical industry.

The major focus in this program under EPA contract 68-02-2153, "Measurements of High-Temperature, High-Pressure Processes," was that of particulate sampling in advanced coal conversion technologies. Those technologies of particular interest were pressurized fluidized bed combustion (PFBC) and coal gasification (CG). Although coal gasification received attention during this program, far more effort was devoted to PFBC.

At present it has become more and more evident that the successful development of a pressurized fluidized bed combined cycle power system hinges on the ability to produce a clean gas stream. The reasons for providing a clean gas stream are two-fold. First, from an environmental point of view, low levels of particulate emissions must be achieved so that limits set by the EPA and state regulatory agencies can be met. Secondly, and more importantly to the power system developer, the hot gas stream must be sufficiently free of particulate matter to allow for long turbine life (References 1 and 2). In general, it cannot be conclusively stated that meeting one criteria automatically satisfies the other in all cases. A lot depends on the required control level of environmental emissions in a given location and the particular design constraints of the turbine in question. Nevertheless, this all points to the fact that high-temperature, high-pressure particulate and gaseous cleanup systems must be developed to provide for high thermodynamic efficiency (by cleaning at elevated temperatures and pressures) and long system life (by maintaining low levels of erosive and corrosive materials). This report focuses predominately on those aspects related to particulate collection.

To develop cleanup systems to meet environmental and turbine standards, it is necessary to perform stream sampling both upstream and downstream of each cleanup stage. In so doing, the removal efficiency of each stage can be determined. The measurements must be accurate and comprehensive. Not only must total stream particulate loading be measured but particle size distribution must be determined. It is well known that the overall efficiency of various particulate cleanup devices is sensitive to the upstream size distribution. It is also well known that the particle size distribution strongly effects the erosion and deposition of turbine components. In addition, the chemical composition of the particulate is important from the standpoint of corrosive reactions that can occur within the cleanup system and on turbine surfaces.

Hence, it is clear that the sampling system must be accurate, reliable, and allow for post examination of the sample. The system should provide the capability to survey across the duct to determine nonuniformities in particulate concentrations and particle size stratification.

The system constraints defined above all point to the selection of an extractive sampling approach for the purpose of developing advanced power systems. *In situ*, laser/optical approaches to particulate measurement are currently under development (References 1 and 3). Most of these methodologies are yet to be proven in high-temperature, high-pressure environments. Once they are fully developed they will be extremely useful tools, but in many applications their primary function will be for monitoring system upsets on an operational power system. In addition, their operation is generally based on the principle of optical light scattering to determine an optical diameter. The more conventional extractive approaches discussed in this report collect and size particulate matter on the basis of particle aerodynamic diameters. This parameter directly correlates to the operational function of inertial and noninertial cleanup devices (cyclones, barrier filters, and granular beds) and the turbine itself. In the case of laser/optical devices, the optical diameter is often difficult to correlate with the aerodynamic particle diameter. Optical devices offer less flexibility for instance, duct surveys are more difficult and no portion of the particulate is available for chemical analysis. Operation of some optical devices depends on maintaining a clean window in the duct. This can be difficult to sustain for long periods of operation.

The arguments delineated previously for the selection of an extractive approach to particulate sampling for the development of PFBC power systems can easily be extended to the development of other coal conversion technologies currently under development such as low-Btu gasification combined power cycle and magnetohydrodynamic (MHD) coal conversion technologies.

1.2 SUMMARY OF WORK PERFORMED

The purpose of the work performed under EPA Contract 68-02-2153 was to assess and develop the technology required to perform high-temperature, high-pressure sampling. Efforts were put forth in several directions. The major emphasis of this program as mentioned earlier, was upon the development of HTHP sampling hardware for coal energy conversion technologies. Other activities included the development of special purpose sampling hardware for coke oven sampling, a versatile probe for sampling conventional/combustion processes at the Research Triangle Park, Environmental Protection Agency, Industrial Environmental Research Laboratory (RTP EPA/IERL) experimental facilities and the high-temperature calibration of series cyclone trains for the EPA Source Assessment Sampler (SASS). A detailed description of these activities can be found in References 4 and 5. Hence, the information in this report concentrates on the findings of those activities related to coal conversion technologies.

The major portion of work performed under this contract included the development of sampling hardware and subsequent testing in a PFBC test rig. These efforts were eventually brought to fruition at the Exxon Miniplant in Linden, New Jersey in March 1977. Two test series were performed at duct conditions of 9 atm and 1350°F. The detailed results of these tests are described in References 6 and 7.

Valuable experience was gained in the design aspects of HTHP sampling equipment and testing procedures. These related to probe design, sampling procedures, and data analysis. The highlights of the information obtained are explained in more detail in other portions of this report.

In addition to the emphasis placed on PFBC sampling, a background study and planning effort was directed toward possible future sampling efforts in a coal gasification facility. A survey was performed to identify likely test sites and determine what sampling requirements and problem areas would be encountered. This, in turn, led to a study of how best to sample gas streams containing tar vapors. Several methods were proposed for separating and collecting these tars from the solid particulate matter present. A design exercise led to a proposed system for sampling in a coal gasification process stream.

Concurrent with the experimental tasks performed at the Exxon Miniplant, a state-of-the-art (SOA) review of HTHP technology was performed. The activities carried out under this portion of the program were also varied. This included a study of practical techniques for measuring stream variables such as pressure, temperature, and velocity. A comprehensive telephone survey was carried out to determine the possible future applications and requirements for HTHP sampling.

The survey also included attention to special sampling problems that might be expected to occur in practice. The highlights of all of the SOA efforts are discussed in other sections of this report.

Other SOA review activities included a detailed study of problems associated with the selection of materials for HTHP probes. Material problems occurring in the reducing atmospheres found in gasification systems present a much more severe problem than those associated with oxidizing environments such as those in fluidized bed combustion applications. Highly corrosive gases, such as H_2S, present at elevated temperatures pose substantial problems to the HTHP probe designer in selecting metals capable of withstanding these exposures. A detailed discussion of these problems and recommended solutions can be found in Reference 4.

Finally as a means of documenting the materials collected, a bibliography of articles, reports, and books relating to HTHP sampling technology and selected coal conversion technologies was assembled and is contained as one of the appendices of this report. In addition, through the various contacts established in the course of the entire project, a mailing list of interested persons was prepared. This mailing list is also contained in one of the appendices of this report.

1.3 GENERAL FINDINGS AND RECOMMENDATIONS

The major effort conducted under this contract was, as mentioned previously, a demonstration of the extractive HTHP sampling approach conducted at the Exxon Miniplant. Test conditions were 9 atm and 1350°F. The major findings of this portion of the program are summarized as follows.

During the first of two test series, it was demonstrated that the PFBC test stream could be successfully contained using a concentric tube sliding seal approach in conjunction with a double gate valve arrangement for sampler isolation while not in use. Procedures for probe insertion, sampling, and withdrawl were successfully demonstrated during the different test runs.

Test gases were cooled using a Dowtherm cooling system to a nominal temperature of 450°F and conventional impactor and filter designs were used. During the demonstration tests, particulate samples were collected both on a series of seven impactor plates (two runs) and also on a thimble filter (one run). Repeatability of test data using the two impactor test runs was judged as good to excellent. Particulate matter was collected during the two impactor runs over a size range of 0.3 μm to 30 m. This material was subsequently examined photomicrographically and analyzed chemically. Results of this analysis yielded no unexpected results.

A second test series was conducted to examine the possible occurrence of alkali metal condensation during sample cool down prior to collection. The sampling probe was reconfigured so that the sample first passed through a scalping cyclone and total filter combination at test stream conditions. Subsequently, the sample was cooled by the Dowtherm cooling system followed by a second total filter. If alkali metal condensation had occurred then it was expected that the downstream filter catch would show a larger alkali metal content. Chemical analysis failed to show any significant alkali metal condensation effects although these limited results were by no means conclusive.

The Exxon results demonstrated that the methods developed and the results produced proved wholly satisfactory for this application. Had the technology been demonstrated at the time, cyclones calibrated for high-temperature, high-pressure operation would have proven more versatile and eliminated the need for cooling prior to collection.

The experience of the Exxon sampling program especially with regard to hardware development and the results of the SOA survey pointed to various conclusions regarding the universality of future sampler designs. Owing to the wide range of test stream conditions including temperature, pressure, gas composition, particulate loading, stream velocity, and duct diameter the impact upon standardization of design is great. It was found that design requirements tend to be nonstandard, thus sampler designs require custom design approaches in response to the particular set of above mentioned parameters.

These broad design requirements inevitably lead to new engineering problems and a consequential custom engineered system. Based on experience gathered during this program, the costs of engineering, designing, calibrating, and acceptance testing of these systems is deemed to be high. As a result, the development costs of one-of-a-kind systems are high with subsequent cost reduction occurring on duplicate systems if they are required.

Some new approaches to HTHP sampling system design were identified. Future systems can conceivably be built at lower cost with greater versatility and safety by using a "total enclosure" design approach in which the traveling probe is totaly isolated, thus eliminating the need for sliding seals. As mentioned previously, it was concluded that HTHP cyclones need to be developed and tested as a means of increasing the allowable sampling time over that of impactors. Where a cooling system may be required to condense water and acid constituents, a water mist approach may prove simpler and less subject to maintenance problems than the Dowtherm system used at Exxon.

In addition to these improvements, new forefronts of research and development were identified. The next challenging problem confronting HTHP sampler developers is that of coal gasification emissions sampling. Problems such as separation and collection of tars, selection of probe materials compatible with corrosive, reducing atmospheres, and explosion hazards must be overcome.

These aforementioned problems are not simple and will be overcome only through diligent research and development programs. It is difficult to foresee the process developers or other members of the private sector bearing the full costs of development for advanced HTHP sampling systems. The burden, therefore, falls on government agencies to provide the necessary research and development funds that are required to bring these systems into common application.

SECTION 2

INTRODUCTION

This section describes the work performed under this program and identifies the near and far term needs for HTHP sampling in the future.

2.1 PROGRAM WORK ACTIVITIES

The primary purpose of the program conducted under EPA Contract 68-02-2153 was to develop techniques and evaluate problems associated with sampling at high temperature and pressure. Strong emphasis was placed upon techniques required for sampling in coal conversion systems such as PFBC and low- and high-Btu CG systems. The results of all contracted efforts are described in References 4, 5, 6, and 7. The paragraphs that follow provide a brief explanation of the various tasks undertaken during this 3-year program. The reader is encouraged to consult the aforementioned references to obtain detailed descriptions of these activities.

2.1.1 PFBC Sampler Hardware Development and Demonstration Tests

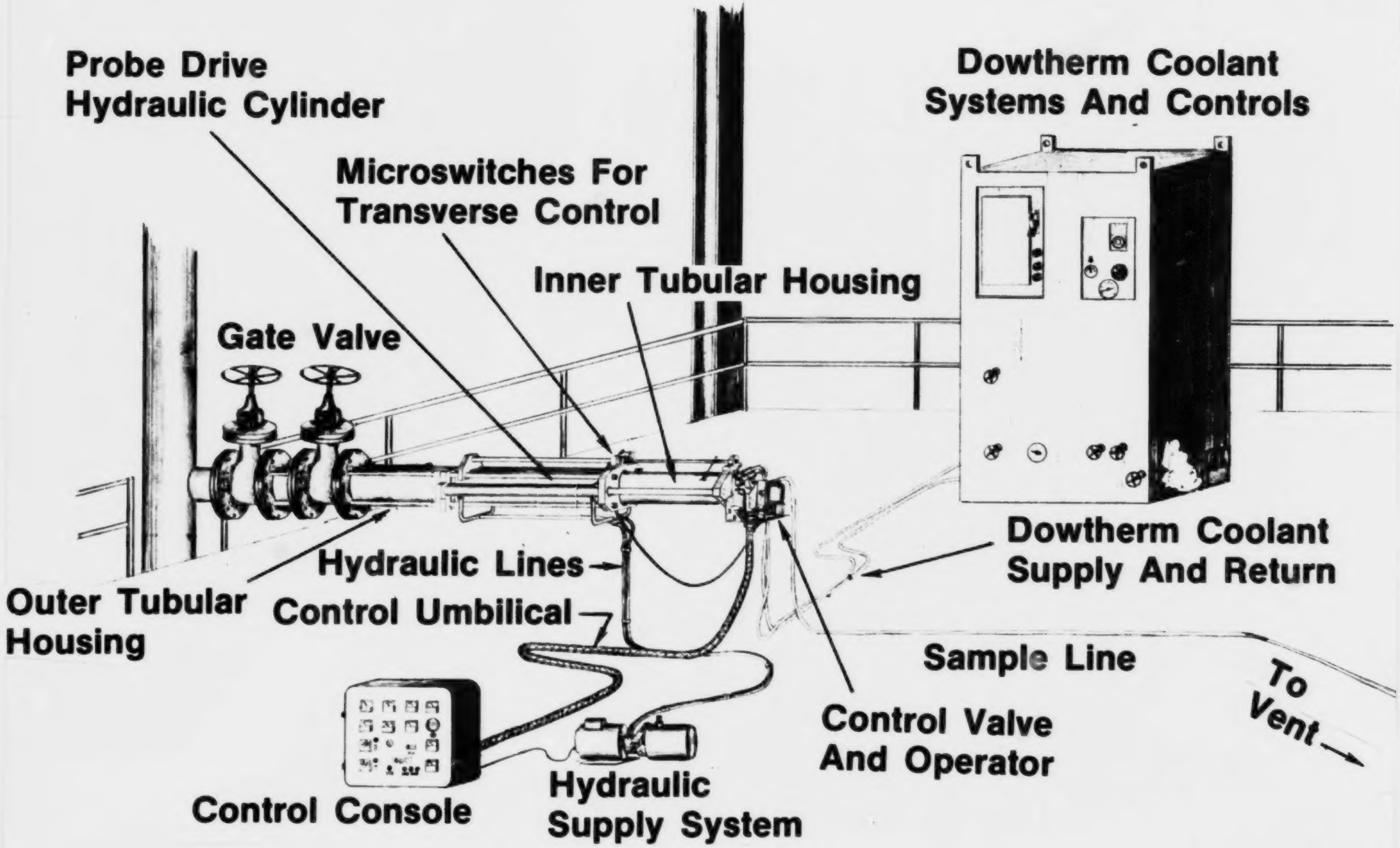
During the period of performance of this program, a major effort was devoted to engineering, designing, and building a prototype sampling system for primary application in a PFBC. The design and engineering of such a system is by no means simple, and constituted a major portion of the efforts put forth by the project team.

Table 1 shows the general capabilities of the PFBC probe designed to operate at the Exxon Miniplant at Linden, New Jersey. The nominal operating conditions at Exxon were 9 atm and 1350°F. The general configuration of the sampling system is shown in Figure 1. Here the following components shown schematically in Figure 2 include:

- The sample probe assembly including the sample collection system (a choice of a seven-stage University of Washington Mark III cascade impactor and final filter or a thimble filter assembly alone)
- A Dowtherm cooling system used to cool the sample to a nominal temperature of 450°F so that a conventional impactor design could be used since no SOA high-temperature, high-pressure collection device was available

TABLE 1. SAMPLING SYSTEM CAPABILITIES

Sample Environment	
• Temperature	650°C - 1000°C (1200°F - 1800°F)
• Pressure	300 - 2000 kPa (3 - 20 atm)
• Gas Constituency CO CO_2 NO SO_2 H_2O NO_x H_2S trace organics trace inorganics	Concentrations subject to further investigation, dependent on process sampled
• Stream Velocity	2 - 46 m/s (8 - 150 fps)
• Particulate Grain Loadings	0 - 34.3 g/m^3 (0 - 15 gr/ft^3) (subject to further consideration and actual process characteristics)
• Particulate Size Range (for classification)	0.2 - 26 microns (Notes: Larger particulates may be acceptable in most cases of total mass determination, or if classified, they may be amenable to "scalping" ahead of classification device)
• Duct Size	Variable depending on probe; std. is 20.3 cm (8 in.) I.D. minimum
Sampling System Configuration	Modular, so as to allow in-situ or extractive sampling by cooled probe
Traverse Capability or Penetration of Nozzle into Duct or Vessel	Approximately 46 cm (18 in.) either in-situ or extractive configuration (some dependence on internal configuration of duct or vessel). Can be extended by relatively minor hardware modification (longer probe, chamber extension, spool piece, etc.)
Access Process Port Requirements	Standard: 10.2 cm (4 in.) IPS minimum, 136 kg (300 lb.) flange access through 10.2 cm (4 in.) IPS alloy gate value (Note: Smaller ports may be acceptable if special probe assembly is used)
Stream Constituency Analysis	Particulates, gases (inorganic and organic, trace elements, trace organics)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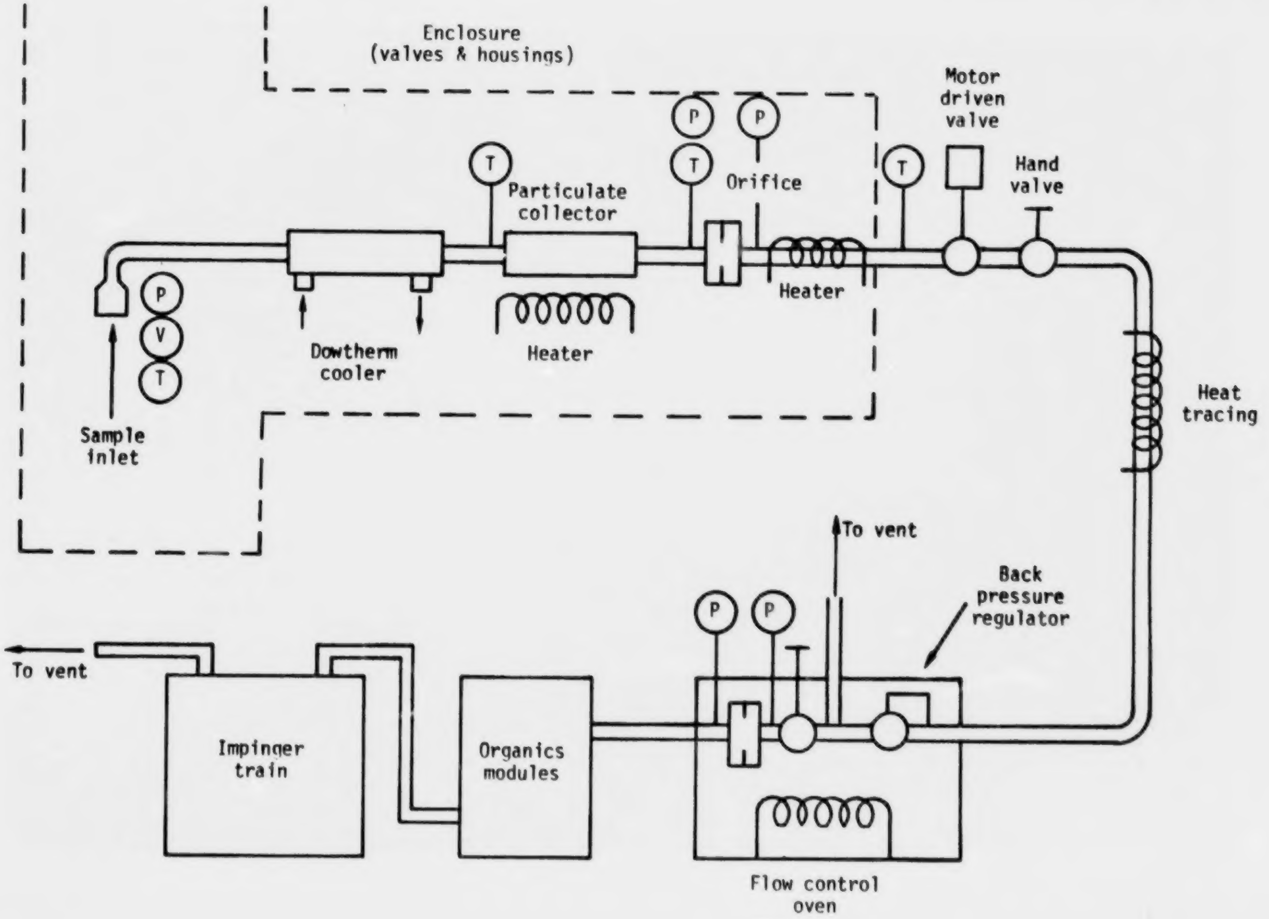
Transverse Control
Inner Tubular Housing
Dowtherm Coolant
Systems And Controls
Gate Valve
Dowtherm Coolant
Supply And Return
Hydraulic Lines
Outer Tubular
Housing
Control Umbilical
Sample Line
To
Vent
Control Valve
And Operator
Hydraulic
Supply System
Control Console

Figure 1. High-temperature, high-pressure (PFBC) sampling system.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Particulate collector
Orifice
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Motor driven valve
Hand valve
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T
Dowtherm cooler
Heater
Heater
Sample inlet
Heat tracing
To vent
Back pressure regulator
P
P
To vent
Impinger train
Organics modules
Flow control oven

Figure 2. System schematic.

- A hydraulic actuator system that was required to overcome the blowoff loads created by use of the concentric traversing tube design (see Section 4)
- A flow control oven that includes orifice and metering valves to maintain the flow at 450°F
- A trace organic module containing a chemical solvent (XAD-2) to remove trace organics present
- Trace metal impinger train that contains selected chemical reactants to condense out various trace species
- Various control consoles to monitor the systems and measure stream and system parameters

The fully assembled probe is depicted in Figure 3.

Field tests were first conducted during March and early April 1977 at the Exxon Miniplant. The sampling location for demonstration purposes was downstream of the secondary cyclone and upstream of the final cleanup device which at that time was proposed to be a Ducon granular bed filter. Test procedures were developed to ensure safety for the operators and good data reproducibility. Standard EPA Level 1 Environmental Assessment cleaning procedures were utilized for the purpose of post-test washing of all probe components to collect deposited materials.

Two test series were conducted. The first test series began in March 1977 with the second series taking place several weeks later after some modified internal parts were fabricated. The purpose of the first series (which consisted of three test runs) was to demonstrate that the general approach taken with respect to stream containment and sample collection was viable. The second test series was conducted to investigate whether alkali metal compounds present in the flyash could have been condensing out of the extracted flow due to the gas stream cooling to 450°F prior to collection. Alkali metal compounds are particularly corrosive to turbine blades.

During the first test series the probe was configured as shown in Figure 4(a) (where a thimble filter could be substituted for the cascade impactor also). In the second test series the probe was configured as shown in Figure 4(b). Here a scalping cyclone and high-temperature filter (saffil alumina) were used to capture the bulk of particulate material present in the test stream. The sample stream containing gaseous components and fines then passed through the cooler and a multiple choke system designed to reduce the pressure while minimizing stream cooling due to supersonic expansion. The sample line was then heat traced before entering a backup filter located external to the main housing. The objective was to chemically analyze the trace metal composition of the high- and low-temperature filters to determine if condensation had occurred. The pertinent results of both test series are discussed in Section 4.5 of this report. The reader is referred to Reference 2 for a detailed discussion of the hardware and the test results.

2.1.2 Gasifier Sampling Site Survey

During the early phases of the program a potential secondary sampling demonstration was identified namely at a pilot-scale coal gasification site. At that point in the program the purpose was to identify particular problems

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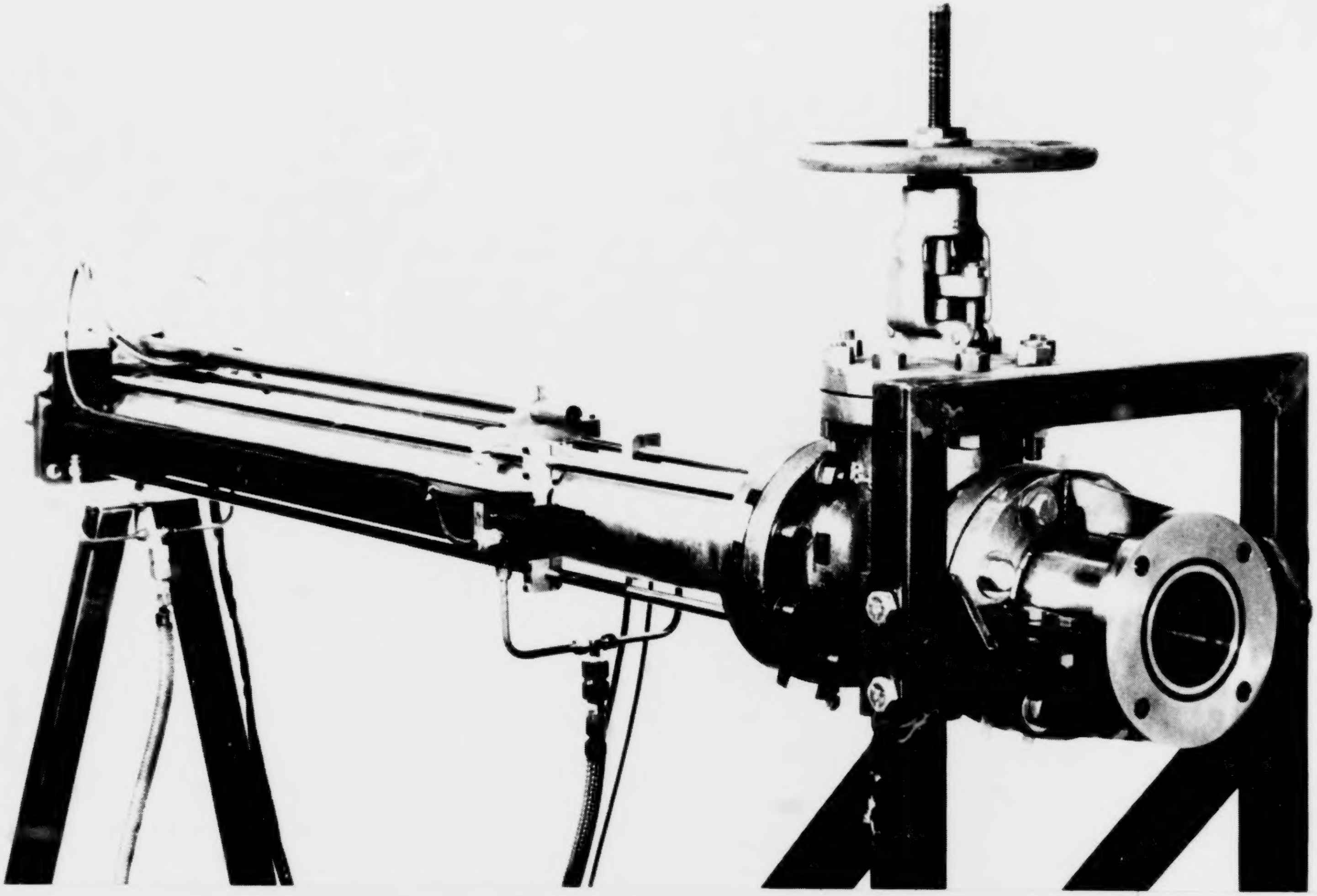

Figure 3. HTHP sampling probe and duct interface valve.

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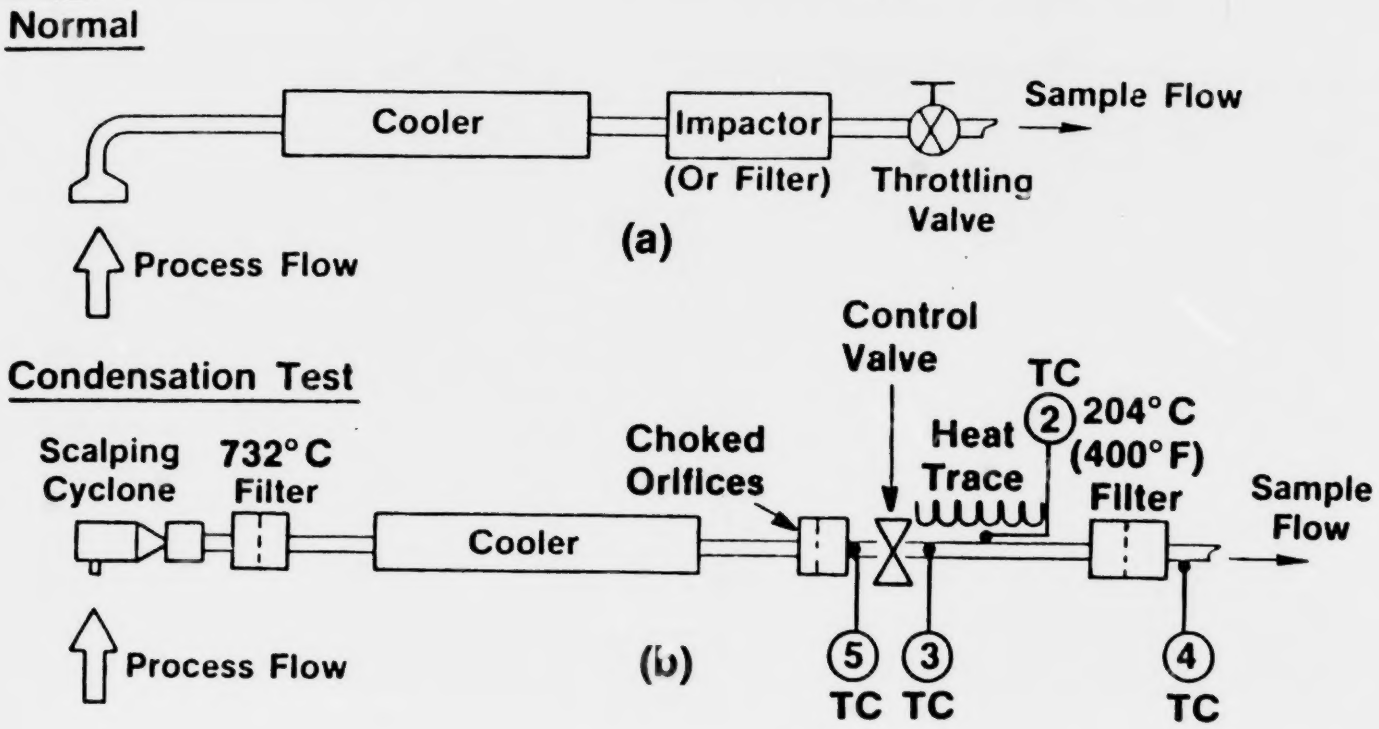
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Impactor
(Or Filter)
Throttling
Valve
Sample Flow
(a)
Process Flow
Condensation Test
Scalping
Cyclone
732°C
Filter
Cooler
Choked
Orifices
Control
Valve
Heat
Trace
TC
2
204°C
(400°F)
Filter
Sample
Flow
Process Flow
(b)
5
TC
3
TC
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TC

Figure 4. Probe configuration.

associated with sampling in a coal gasification facility. It was conceived at that time that if sampling requirements could be met with a new or modified probe, preparations for gasification sampling could be started under this program.

A site survey as well as a telephone survey was conducted to identify potential sampling locations, determine what the sampling requirements are, and to identify what special problems require solutions. After the survey was completed a work plan was prepared outlining a proposed program for sampling at alternative sites yet to be determined. Several potential sites suggested are shown in Table 2.

Upon completion of the survey it became evident that the scope of a complete program for building a gasifier sampling probe and conducting a sampling demonstration test would be beyond the funds available under the existing program. The problems identified with sampling at a gasifier require a major engineering and design effort to construct a new probe system. A major problem in terms of sampling at a gasifier is that of developing a means of condensing vaporous tars for analysis while keeping them separate from the particulate material. Other problems peculiar to sampling in a gasifier include the selection of materials that will be compatible with a highly corrosive, reducing atmosphere and that of producing an explosion-proof sampling system.

Since such an air ambitious program was not possible within the funds available, it was decided that two of the problem areas be addressed under the SOA review portion of this program. A study was conducted to explore various means of collecting and separating tars from a gasification stream. Some of these findings are discussed elsewhere in this report. A second effort undertaken in the SOA review was that of examining the problems of material compatibility in a gasification environment. This study was performed from a metallurgist point of view. The complete findings of both of these studies are found in Reference 4.

2.1.3 Source Assessment Sampling System Development (SASS)

A development task was undertaken to make improvements to the EPA's recognized Level 1 Environmental Source Assessment Sampling System, the SASS. Several modifications were made that gave improved performance, better maintainability, and extended cyclone operating range. The aspect that most related to HTHP technology was that of the development of a calibration test facility designed to calibrate cyclones at elevated temperatures up to 400°F. A rotating dust table, dust pickup mechanism, and dispersion system was conceived and developed under this task. A standard SASS cyclone train consisting of three cyclones was calibrated at a temperature of 400°F. A schematic of the test rig is shown in Figure 5. The complete details of this effort are discussed in References 4 and 5.

2.1.4 Special Purpose Probes

Two special purpose probes were constructed for particular applications. Both of these probes were based upon the basic SASS design and were modified

TABLE 2. POTENTIAL GASIFIER SAMPLING SITES

Category	Facility	Developers/Sponsors	Gasifier Type	Temperature (F)	Pressure (psia)
1.	$P<300$ psia, $T<1800$ F, gas composition relatively low tar concentrations				
	● Westinghouse Waltz Mill	ERDA	Multiple fluid beds with dolomite regenerator step	1400-1800	150-240
	● CO_2 Acceptor	Consolidation Coal CO./ERDA, AGA	Fluidized bed using dolomite/limestone as acceptor for CO_2 and H_2S	1500-1550	150-300
2.	$P<300$ psia, $T<1800$ F, higher tar, particulate, sulfur loading in product gas				
	● Stirred bed	U.S. Bureau of Mines	Fixed bed reactor with agitator	1000	300
	● G.E. Gas Schenectady, N.Y.	G.E./EPRI	Moving fixed bed reactor with countercurrent flow	1000-1100	170-350
	● Union Carbide Ash Agglomeration	Battelle/ERDA	Two fluidized beds	1600-1800	120
3.	$P>300$, $T<1800$				
	● Bi-Gas Homer City	Bituminous Coal Research/OCR-AGA	Two-stage entrained beds, upflow configurations	1800	1000-1500
	● Hi-Gas	IGT/ERDA	Two-stage fluid bed hydro-gasifier; electrothermal/oxygen/steam-iron/steam-air bed char gasifier for H_2	600-1200	1000-1500
	● Synthane	U.S. Bureau of Mines	Fluid bed reactor	1100	500-1000
	● Grand Forks Energy Research Center	ERDA	Lurgi-slagging design	400-1500	400
4.	$P>300$, $T>1800$				
	● U-Gas	IGT/ERDA	Fluidized bed reactor, ash-agglomerating gasifier	1900	300-350

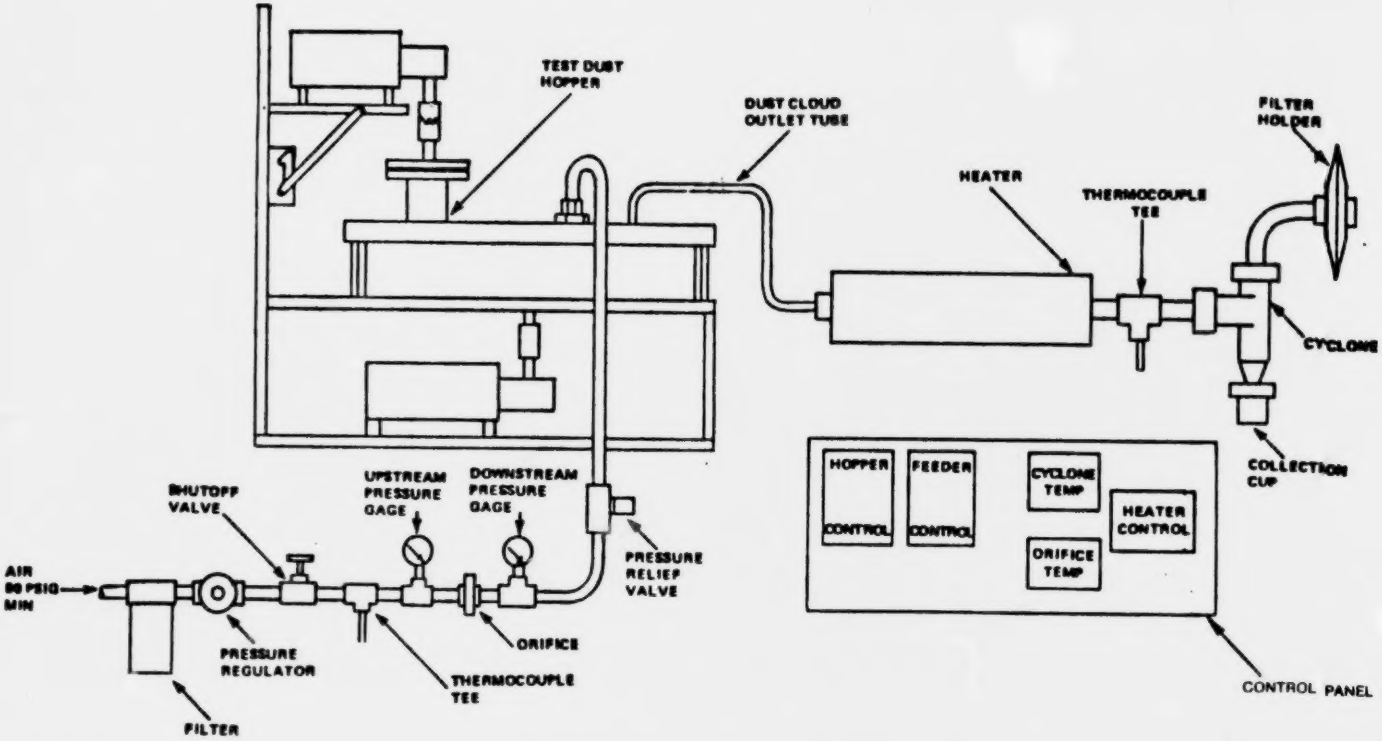
TEST DUST HOPPER
DUST CLOUD OUTLET TUBE
HEATER
THERMOCOUPLE TEE
FILTER HOLDER
CYCLONE
COLLECTION CUP
HOPPER CONTROL
FEEDER CONTROL
CYCLONE TEMP
ORIFICE TEMP
HEATER CONTROL
CONTROL PANEL
AIR 80 PSIG MIN
SHUTOFF VALVE
UPSTREAM PRESSURE GAGE
DOWNSTREAM PRESSURE GAGE
PRESSURE RELIEF VALVE
PRESSURE REGULATOR
ORIFICE
THERMOCOUPLE TEE
FILTER

Figure 5. Cyclone test apparatus.

to operate at elevated temperatures. Both probe designs utilized a water cooled jacket to maintain probe integrity. One of the probes shown in Figure 6 was designed to operate in a variety of applications on experimental combustion facilities located at the EPA Industrial Environmental Research Laboratories at Research Triangle Park, North Carolina. A description of the probe itself and operational procedures are contained in Reference 4.

The second probe was of a similar but simplified water-cooled design and was specifically developed to evaluate the environmental pollutants emanating from coke ovens during open door conditions.

2.1.5 State-of-the-Art (SOA) Review

Whereas the primary focus of the overall program was upon the PFBC sampling effort, the secondary emphasis was placed upon the SOA review. The major elements of the SOA review included:

- A survey of facilities to determine near and far term HTHP sampling needs
- A study of techniques for measuring pressure, temperature, and velocity in a particle laden stream
- A study of means for collecting and separating tars and solid particulate in a coal gasification stream
- A survey of problems related to selection of materials compatible with coal conversion environments
- A bibliography of information relating to HTHP sampling system technology

Most of the detailed findings for the second, third, and fourth studies are contained in Reference 4. Some aspects, particularly related to tar separation, are updated in this report. The results of the facility survey are contained in Section 3 of this report.

In summation, the various elements that comprise the entire program were very comprehensive in nature. The major thrust of the program was directed toward PFBC probe development and field demonstration and comprised about 60 percent of the total effort.

2.2 NEAR AND FAR TERM NEEDS FOR HTHP SAMPLING

There is a current need for sophisticated extractive particulate sampling equipment. Because this equipment is not generally available the required sampling is accomplished using modifications of equipment designed for conventional environments and through simplified "homebuilt" devices which may not yield accurate results. These approaches can provide order-of-magnitude results that may be adequate for obtaining preliminary data but will not serve for the more refined development of coal conversion technologies that will be attempted soon.

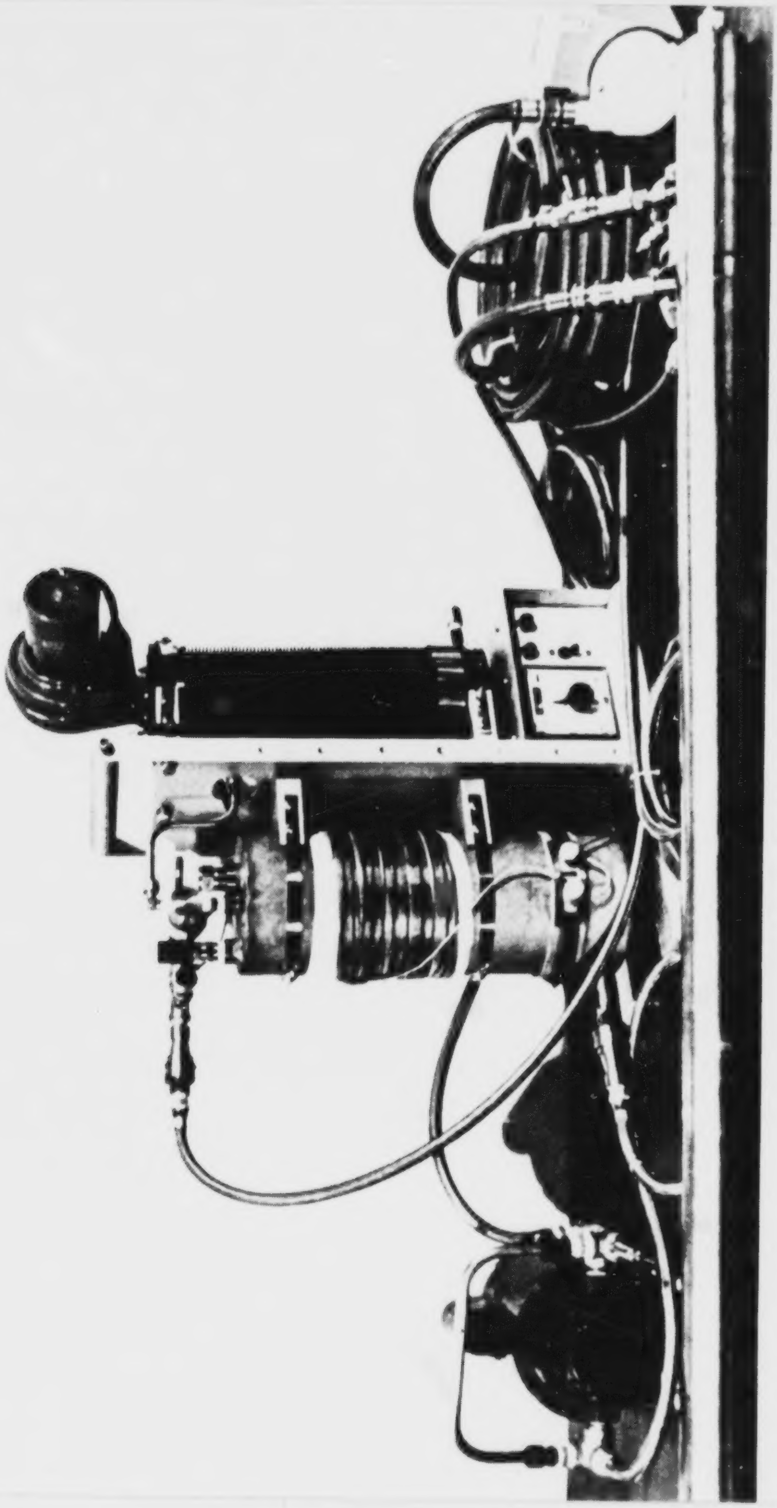

Figure 6. Liquid cooled sampling probe.

The primary reason the required sampling equipment is not currently available is that developing precision equipment for application in the severe environments of the coal combustion zone is extremely expensive. Furthermore, applications of this sampling equipment are generally limited to those few test sites doing research on advanced coal conversion processes. Thus, there is no viable current market for the equipment. For these reasons industry will not sponsor development and, therefore, if it is to be available its development will have to be sponsored by the government.

2.2.1 Pressurized Fluidized Bed Combustion

One of the most significant application areas for HTHP extractive particle sampling equipment is associated with the development of pressurized fluidized bed combined cycle powerplants. Potentially the process can utilize a wide variety of coal types at high thermodynamic efficiency (40 percent) and do so while controlling SO_x and NO_x without expensive add-on devices.

Major test facilities operating research PFBC systems are the CURL facility in Leatherhead, England, the EPA/Exxon Miniplant in Linden, New Jersey and the PFBC installed at Curtiss Wright Corporation in Woodbridge, New Jersey. A new and larger facility is being built under International Energy Association (IEA) sponsorship at Grimethorpe in England. American Electric Power Corporation, in conjunction with Stal Laval, a Swedish turbine manufacturer, is preparing preliminary designs for a utility-sized PFBC to be built in Ohio. Each of these facilities will have continuing needs for reliable extractive particle sampling equipment as process development continues and the hot gas cleaning problem is addressed over the next several years.

In the combined cycle process, coal is burned in a pressurized, fluidized bed of sorbent material such as dolomite or limestone. The bed is fluidized by injecting compressed air under and through the bed. The dolomite or limestone is injected into the bed in sufficient quantity to react with the sulfur gases released through this combustion of coal. Steam tubes are located within the bed and water is passed through these tubes at a rate such that the combustion temperature is controlled at 800 to 900°C. This relatively low combustion temperature limits production of thermal NO_x.

Most proposed PFBC systems are designed to operate at between 10 and 20 atm of pressure. The hot, high-pressure gases leaving the bed contain the gaseous combustion products and large quantities of particulate flyash. This HTHP gas stream is expanded across a gas turbine to drive the compressor and generate electrical power in the second of the two cycles comprising the combined cycle system. Development of methods to protect the gas turbine from particulate erosion and corrosion represent the major tasks required before commercialization of this advanced coal combustion technology can take place. Reference 1 discusses the levels of cleanup required by various turbine manufacturers.

To more fully understand the system, particle sampling is needed at several locations within the process. These locations include sampling within the bed, before and after one or more stages of cyclone separation, upstream and downstream the fine particle removal device, and downstream of the turbine. Commercially available low-temperature, low-pressure equipment is suitable only for sampling downstream of the turbine.

The high temperatures and pressures combined with erosive and corrosive contaminants present in the operating environment create severe design and materials limitations to the design of the sampling system. These factors create problems in construction, sealing, cooling, and require the use of high performance materials. Major design constraints for an HTHP particle sampler are listed:

- Severe environment -- Temperature, pressure, corrosives
- Flow stream problems -- Tortuous paths
- Confined space -- Limits work space
- Variation in particle concentration -- Affects sampling time and capacity
- Safety -- For operators
- Cost -- Must be affordable

As a result of the high pressure and temperature conditions, gas distribution piping is costly and long straight sections ideal for particulate sampling are seldom available. In addition, the sampling locations are often cluttered with other equipment, structures, and piping. These conditions require that the sampler be capable of isokinetic operation while also being compact so that it can be installed where needed. For isokinetic sampling at locations with a less than ideal flow distribution, the sampler must have the capability to sense and be adjusted to changes in flow velocity and must posses a high degree of transverse and angular mobility.

At the locations where sampling is needed, the particle mass concentration can vary over several orders of magnitude. This means that the sample quantities collected, the collection time, and the volume sampled or a combination of these parameters must be capable of being varied if a single design is to be used at different locations.

A gas leak failure of the system could pose a potentially lethal hazard to nearby operating personnel. For this reason, the sampler must be rugged, reliable, and be designed with operator safety as a major consideration. It is highly desirable, if not mandatory, that the sampler be capable of remote operation. This is probably the least expensive and most positive way to ensure operator safety.

Cost is another design constraint. A review of the technical constraints above should provide some understanding of why estimated costs of this equipment have been high. This is especially true when the order quantities are limited to one or two prototypes. As a result of several design efforts to date at Acurex, the predicted costs for HTHP samplers have been reduced. These reductions are a result of refinements in design concepts leading toward simplification of hardware. Once the initial development costs are completed and the design concept is demonstrated, additional copies of the most simple probes should find application in other PFBC facilities at costs only somewhat above those of more conventional sampling equipment.

2.2.2 Coal Gasification Sampling Development

Coal gasification is basically an old technology that is receiving renewed attention as an efficient means of extracting a gaseous fuel from coal and using it either to the site of gasification or transporting the gas via pipeline to be burned offsite. Gaseous fuel burned onsite in turbines or boilers is usually of the low-Btu variety (100 to 300 Btu/scf) while high-Btu gas (800 to 1000 Btu/scf) produced through a methanization process would be used as a substitute pipeline fuel for natural gas.

The basic differences between the two process types is governed by the operating temperatures and pressures and by whether the gasification bed is air blown or oxygen blown. High-Btu gasification processes tend to be oxygen blown and operate at extremely high temperatures and pressures. Reference 8 presents the significant features of more than 20 operating coal gasification processes located in the U.S. Table 3, taken from Reference 9, presents a comparision of 19 coal gasification processes. The typical operating temperatures and pressures for each facility are shown. The operational ranges for gasification system dictate the need for high-temperature, high-pressure or at the least, high-temperature sampling approaches.

Operating pressures for some high-Btu gasification processes are enormous even compared to PFBC sampling applications and dictate that special means of pressure containment be used. For example, it is highly unlikely that a pressure housing such as that used at the Exxon PFBC, (concentric tubes separated by a sliding seal) be applicable at pressures as high as 1200 psi. Sealing requirements would be untenable in any event. Also, the blowoff loads would be extreme and would require a very large hydraulic actuation system to traverse the probe. Alternatives to these approaches are discussed in Section 4 of this report.

The needs for using in process HTHP particulate sampling systems in gasification processes with which the authors of this report concur are identified by the authors of Reference 8. They are as follows:

- To measure particulate carryover from the reactor bed
- To determine the chemical composition of the particulate leaving the bed
- To determine the concentration of mositure, tars, trace elements, etc., at various points in the process
- To determine particulate levels prior to the methanization step (for high-Btu applications) to prevent catalyst poisoning
- To determine particulate level and composition upstream of the turbines (for low-Btu applications)
- To evaluate the performance and aid in the design of particulate cleanup devices

Many of the above needs and requirements are shared with PFBC applications. One requirement that is unique to sampling applications in coal gasifiers is that of sampling tars.

TABLE 3. SUMMARY OF COAL GASIFICATION PROCESSES

IDENTIFIER	LOCATION	TYPE OF BED	TEMP. °F	PRESS. PSIG	AIR OR O_2	I.D. FT	COAL TON/DAY	O_2/AIR LB/HR	STEAM TON/HR	SAMPLING	OPERATING
BATTELLE	Richland, Wash.	Fixed	1500	5	Air	3	-	-	-	Impactor	Yes
GLENGERY	Shoemakersville, Pa.	Fixed	1000	1	Air	10	24	-	-	No	Yes
WILPUTTE	Kingsport, Tenn.	Fixed	1200	15	Air	-	-	-	-	No	Yes
NL&S	Carey, Ohio	Fixed	1050	1	Air	-	-	-	-	Only to Satisfy EPA	Yes
R-M	Worcester, Mass.	Fixed	1050	1.5	Both	10.5	90	11.5 T/Hr	1.35	From Cyclone	No
MERC	Morgantown, W.Va.	Fixed	1200	125	Air	3.5	27	3037	0.25	High Temperature Filter	Yes
GE	Schenectady, N.Y.	Fixed	1200	300	Air	3	-	-	-	No	Yes
IGT	Chicago, Ill.	Fluidized	1200	1000	O_2	2.5	72	1720	0.21	Grab Sample	Yes
FMC	Princetown, N.J.	Fluidized	1500	30	Air	6	2.5	-	-	No	Yes
BATTELLE	Columbus, Ohio	Fluidized	1800	100	Air	3	25	-	-	No	Yes
BCR	Monroeville, Pa.	Fluidized	1800	300	Both	-	-	-	-	Planning	Yes
WEST	Madison, Pa.	Fluidized	1750	175	Air	3	15	-	-	Yes	Yes
PERC	Brucetown, Pa.	Fluidized	1750	1000	O_2	5	75	-	-	From Scrubber	Mid '76
CONOCO	Rapid City, S.D.	Fluidized	1500	150	(Air)	3.3	30	0	1.6	From Cyclone	Yes
B&W	Alliance, Ohio	Entrained	1700	35	Both	9	850	30 T/Hr	30	Tried, Filter Plugged with Tar	Yes
CE	Windsor, Conn.	Entrained	1700	15	Air	33	2840	372 T/Hr	35	No	Designing
OCCIDENTAL	Laverne, Cal.	Entrained	1500	50	Air	1	-	-	-	No	Yes
TEXACO	Monte Bello, Cal.	Entrained	1500	300	O_2	2	-	-	-	No	Yes
BCR	Homer City, Pa.	Entrained	1750	1200	O_2	9	-	-	-	Planning	Late '76

During the gasification process trapped volatile materials in the coal is released. Foremost among the variables affecting the quantities of tars and other volatiles released are the operating pressure and temperature and the coal type. Table 4 shows the relative quantities of tars and ash present in four different coal types. Also as shown in Figure 7 the tar yield is directly related to the percentage of volatiles present. It has also been shown that in practice prolonged exposure to high temperatures in the bed increases the tar yield. Table 5 shows the relative yield of gas, volatiles, and solids that are produced in several operating gasifiers. Acurex conducted a separate survey of gasification facilities to determine process development problems relating to tar production. These are discussed in further detail in Section 3 of this report.

Basically, tars in the off gas present two major problems. First, as system pressures and temperatures drop the tars may condense on the walls of the process ducts or clog turbine passages. In addition, the tars themselves present a health hazard to those who are exposed, whether at the process plant or through pipeline application. These tars are known to be highly carcinogenic and breathing their vapors or skin contact should be avoided entirely. On the positive side, these tars are combustible; hence, their energy content cannot be ignored.

Consequently, the presence of tars in the gasification process requires measuring systems to identify their relative quantities and properties within the process. At the same time, the process developer needs to understand the quantities and size distributions of particulate to satisfy the needs and requirements stated earlier. These simultaneous requirements demand that a HTHP sampling system designed for gasifier application have the capability of collecting and separating tars and particulates simultaneously. This is a difficult requirement to fulfill in that throughout most of the gasification process the tars are in a vapor phase. This means that if the tars are in a vapor phase in the process duct they must first be condensed into a liquid phase within the sampler. Unfortunately, the tars present in coal gasification processes are very complex in molecular structure and do not possess a single condensation temperature. Table 6 shows a percentage breakdown of tars from the Grand Forks gasifier. Bear in mind that these are broad classifications and that literally thousands of different molecular species are present.

Condensation of the tar vapors forms a tar mist of very fine particles. The condensation of tar mist must take place downstream of the solid particulate collection device otherwise one will obtain clogging in the probe cyclones and a resultant catch which is a combination of tars and solid particulates.

The proposed sequence of events that must take place within the probe are shown in Figure 8. Here the solid particles are collected at the system temperature and pressure so that the tars remain in a vapor phase. After the solid particulate is removed the temperature is reduced below the condensation temperature of the lowest fractions to be collected.

TABLE 4. TYPICAL COAL AND GAS ANALYSIS

Coal rank	Anthracite		Low vol. A bituminous	High vol. A bituminous	High vol. C bituminous	
Origin	Pennsylvania		Pocahontas	W. Virginia	Ohio	
Dry coal analysis, wt, %						
C	85.5		80.5	83.8	67.0	
N	2.0		4.1	5.2	4.7	
O	1.7		2.8	4.1	6.3	
H	0.9		1.0	1.5	1.1	
S	0.7		0.7	0.7	4.0	
Ash	9.2		10.9	4.7	16.9	
Volatile matter	4.8		16.4	30.1	37.1	
Fired carbon	86.0		72.7	65.2	46.0	
Heating value Btu/lb	13,450		13,850	14,855	12,195	
Free swelling Index	0		3	8	4	
Coal ash softening Temp, ^{o}F	+2,700		--	+2,700	2,360	
Typical gas analysis	Air	O_2	Air	Air	Air	O_2
CO	24.1	38.4	25.4	25.6	25.4	39.2
N_2	18.4	41.9	14.6	13.8	12.3	31.4
CO_2	6.6	18.6	6.8	6.4	4.6	16.2
CH_4	0.5	0.7	0.9	1.8	1.7	3.3
Higher H/C	0.5	--	0.1	0.3	0.1	0.8
H_2S	0.1	--	0.11	0.12	0.30	1.1
NH_3	--	--	0.27	0.05	0.05	0.1
N_2 + inerts	50.3	0.4	51.8	51.9	55.3	7.9
HHV. Btu/Scf	142	266	141	152	139	286

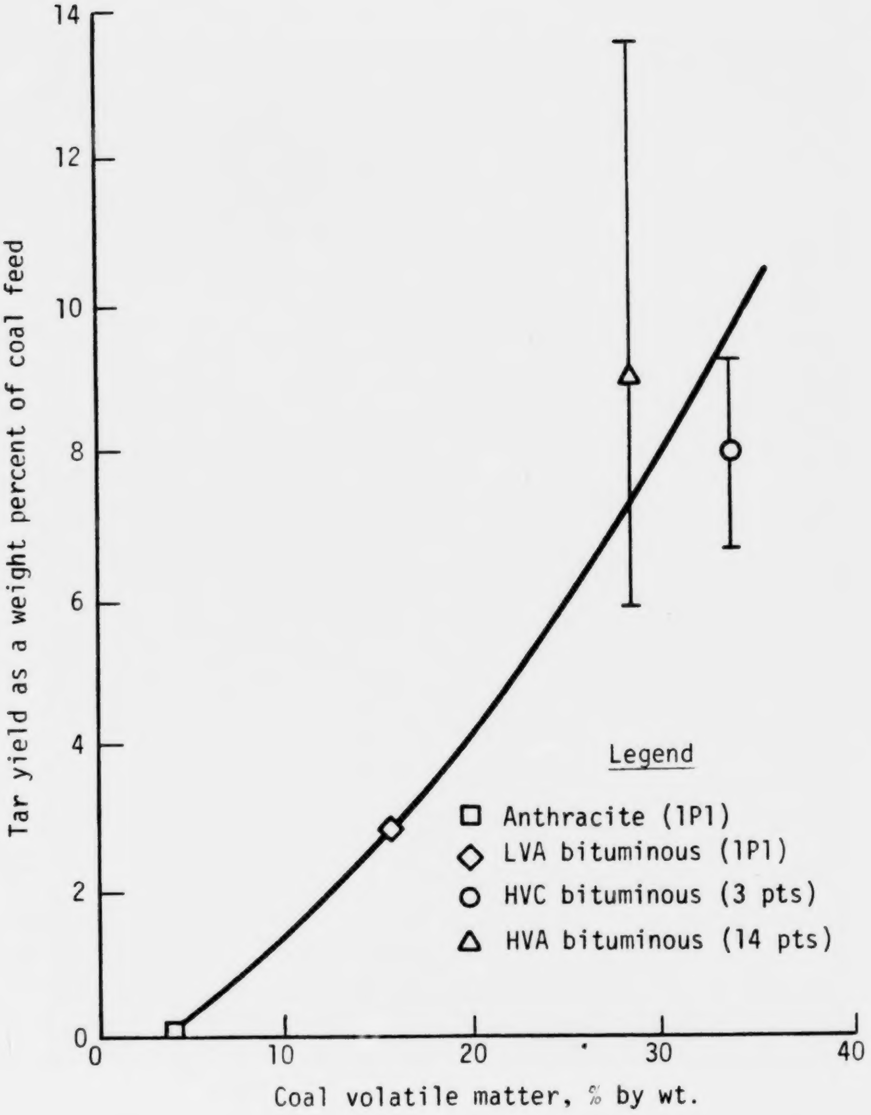

Figure 7. Tar yield as a function of coal volatile matter -- pilot gas producer.

TABLE 5. PRIMARY CONVERTERS -- DISTRIBUTION OF ENERGY INTO PRODUCTS AND BYPRODUCTS

	Percent of Output Energy		
Process	Gas	Tars & Oils	Chars, Ash, & Residue
Koppers-Totzek	95	--	5
Texaco	95	--	5
U-GAS	96	--	4
BI-GAS	95	--	5
CO_2-Acceptor	94	--	6
HYGAS-Oxygen	95	3	2
Synthane	71	4	25
Westinghouse	96	--	4
Union Carbide/Battelle	96	--	4
Winkler	80	--	20
Wellman-Galusha	72	18	10
Lurgi	69	16	15
COED	25	20	55
TOSCOAL	6	18	76
Garrett	35	15	60
H-COAL	10	75	15
CSF	10	75	5
SCR	20	55	25

TABLE 6. GRAND FORKS SLAGGING LURGI TAR ANALYSIS[a]

Constituents	Pct
Saturated hydrocarbon	10.3
Nonvolatile residue	7.7
Phenol/cresol/xylenol	22.4
Indanol	1.0
Dibenzofuran	3.78
Hydroxyanthracene	1.01
Indanes	0.22
Naphthol	3.93
Indenes	0.96
Pyridines	1.81
Quinolines	1.35
Naphthalene	4.76
Acenaphthene/biphenyl	0.37
Fluorene/acenaphthalene	1.76
Phenanthrene/anthracene	0.85
Dihydropyrene	0.81
Pyrene/fluoranthene	0.49
Chrysene	0.49
Benzenes	1.78
Indole	0.23
Carbazole	0.21
Benzocarbazole	0
Benzofuran	5.16
Benzonapthofuran	2.30

[a] Performed by mass spectrometry

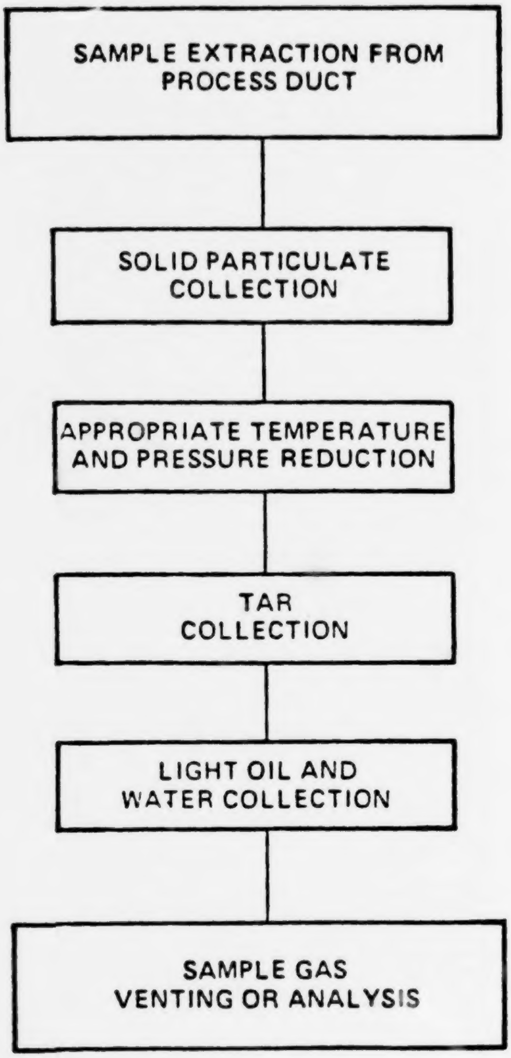
SAMPLE EXTRACTION FROM PROCESS DUCT
SOLID PARTICULATE COLLECTION
APPROPRIATE TEMPERATURE AND PRESSURE REDUCTION
TAR COLLECTION
LIGHT OIL AND WATER COLLECTION
SAMPLE GAS VENTING OR ANALYSIS

Figure 8. Gasification process sampling scheme.

This forms a fine tar that is difficult to coalesce by impingement or other inertial approaches (see Reference 4). Consequently, other means must be used to collect the tars. Several approaches to tar collection were studied during this program. A detailed discussion of these methods is presented in Reference 4. A further discussion of these approaches is also contained in Section 5 of this report. These approaches included:

- Inertial separators
- Filters
- Electrostatic precipitators
- Packed beds
- Scrubbers

The selection and development of one of these tar collection devices is critical to the development of a HTHP sampling system for gasification application. The suggested approach (recommended by University of California at Berkeley researchers) is to use a packed bed of inert ceramic material filled with pyridine, $CH(CHCH)_2N$, as a tar solvent. The pyridine can later be evaporated away. Several collection schemes seem viable using both an ESP and/or a packed solvent bed. Figure 9 shows three possible tar collection schemes. The configuration upstream of the tar collectors is the same in all three cases. It consists of collecting the particulate at high temperature and pressure and then rapidly chilling the gas to condense the tar vapors to form a mist. Chilling to a point just above the condensation temperature for water (at the local P) should be sufficient. Scheme 1 uses a packed bed scrubber in series with an ESP to collect the tars. This arrangement presumes that a packed bed will not be able to remove all the submicron tar mist. Thus, the ESP design effort will be simpler since the operating regime is confined to submicron particles. The filter downstream will collect any residual materials. The gas can then be passed through an impinger train (not shown) to remove any condensibles not removed upstream such as light oils. In Scheme 2 the ESP is replaced entirely by the packed bed. Finally, Scheme 3 shows the ESP as the primary collection device and would be used along only if the packed bed approach proved ineffective.

Other problems that must be overcome in developing the entire system are highlighted in Table 7. Finally, to illustrate typical sampling conditions Figures 10 and 11 and Tables 8 and 9 are presented. These figures and tables present the operating conditions and required sampling locations at the Westinghouse fluidized bed gasification process and the DOE Morgantown stirred bed gasification system.

In summary, there exists the requirement to develop gasification sampling systems and difficult development problems lie ahead.

2.2.3 Other HTHP Particulate Sampling Needs

Applications for special purpose high-temperature, high-pressure samplers exist in a few areas other than PFBC and gasifiers. Two applications recently identified were sampling in the exhaust of an MHD channel and an application at a catalytic cracking plant to determine interstage cyclone efficiency

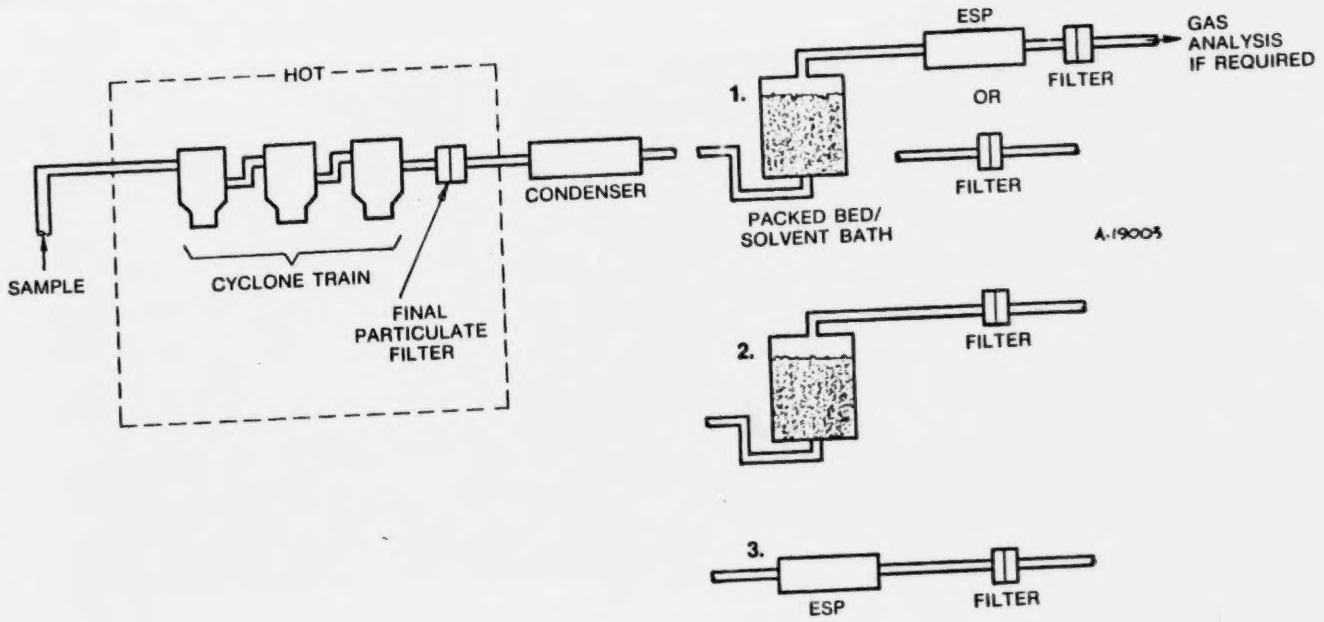
HOT
SAMPLE
CYCLONE TRAIN
FINAL PARTICULATE FILTER
CONDENSER
1.
PACKED BED/ SOLVENT BATH
ESP
FILTER
GAS ANALYSIS IF REQUIRED
OR
FILTER
A-19005
2.
FILTER
3.
ESP
FILTER

Figure 9. Alternative approaches to tar collection for gasification sampling system.

TABLE 7. DEVELOPMENT PROBLEMS OF HTHP GASIFICATION SAMPLERS

Problem	Reason	Solution
• Cyclones must be operated at high temperature and pressure (1000°F, 25 atm typ.)	Particulate removal must be performed initially. Temperature and pressure reductions allow heavy HC species to condense	Smaller actual flow rate requirements (due to increased pressure allow smaller cyclone sizes. Cyclone train can be made integral to the probe body
• Cyclone size variations with temperature, pressure, flowrate	Require a known cut size for modified cyclone designs	Calculations available to demonstrate cut size variation with pressure (none), temperature (small), and flowrate (moderate)
• Isokinetic sampling	Isokinetics must be maintained for particulate sampling	Isokinetics achieved as in present system with change of probe nozzle
• Cyclone accessability	Cyclone system integral to the probe must be accessible for disassembly/sample collection	Provisions made for probe disassembly and cyclone removal. Time requirements are not critical
• Maintain pressurized environment exterior to cyclone bodies	Avoid large pressure differtial between outside and inside of cyclones	Utilize exterior pressure system or pressure equalization line to sample source
• Maintain cyclone train at high temperature (1000°F)	Avoid condensation of heavy hydrocarbon species (tars) in cyclones	Requires possible heating/cooling. Electrical heating probably most applicable in contained pressurized environment
• Collection of heavy hydrocarbon species (tars)	Collection must be performed prior to XAD-2 adsorption	Collection by temperature reduction at high pressure. Efficient collection chamber design is required
• Sample pressure, temperature reduction prior to adsorption cartridge	XAD-2 must be operated at 20°C, atmospheric pressure	Throttle valve, pressure regulator, critical orifice utilized for pressure reduction. Temperature reduction by oven and gas cooler
• Flowrate control	Control gas flows to cyclones and absorber/impinger components	Utilize flow control valve and critical orifice in controlled temperature environment
• Determination of actual cyclone and sample gas flow rates	Maintain proper cyclone flowrates. Knowledge of total sample volume	Use of orifices and/or gas flow meter with temperature and pressure measurement taken to compute other system location flowrates
• Sample transport	Maintain sample flow through the system	Source pressure adequate — no pumping required
• Coolant requirements	Maintain probe structural integrity	Coolant circulation in probe body may be required
• Variations in impinger flowrate	May affect chemical absorption efficiency	Impinger bottle nozzle size variation may be required
• In-probe catalytic effects	Maintain sample chemical integrity between process extraction and final analysis	Study warranted to determine probe/sample interactions at HTHP
• Corrosion, erosion at high temperature and pressure	Effects on final design may be different than at lower temperatures and pressures	Choose appropriate materials to alleviate corrosion. Analyze erosion problems at HTHP

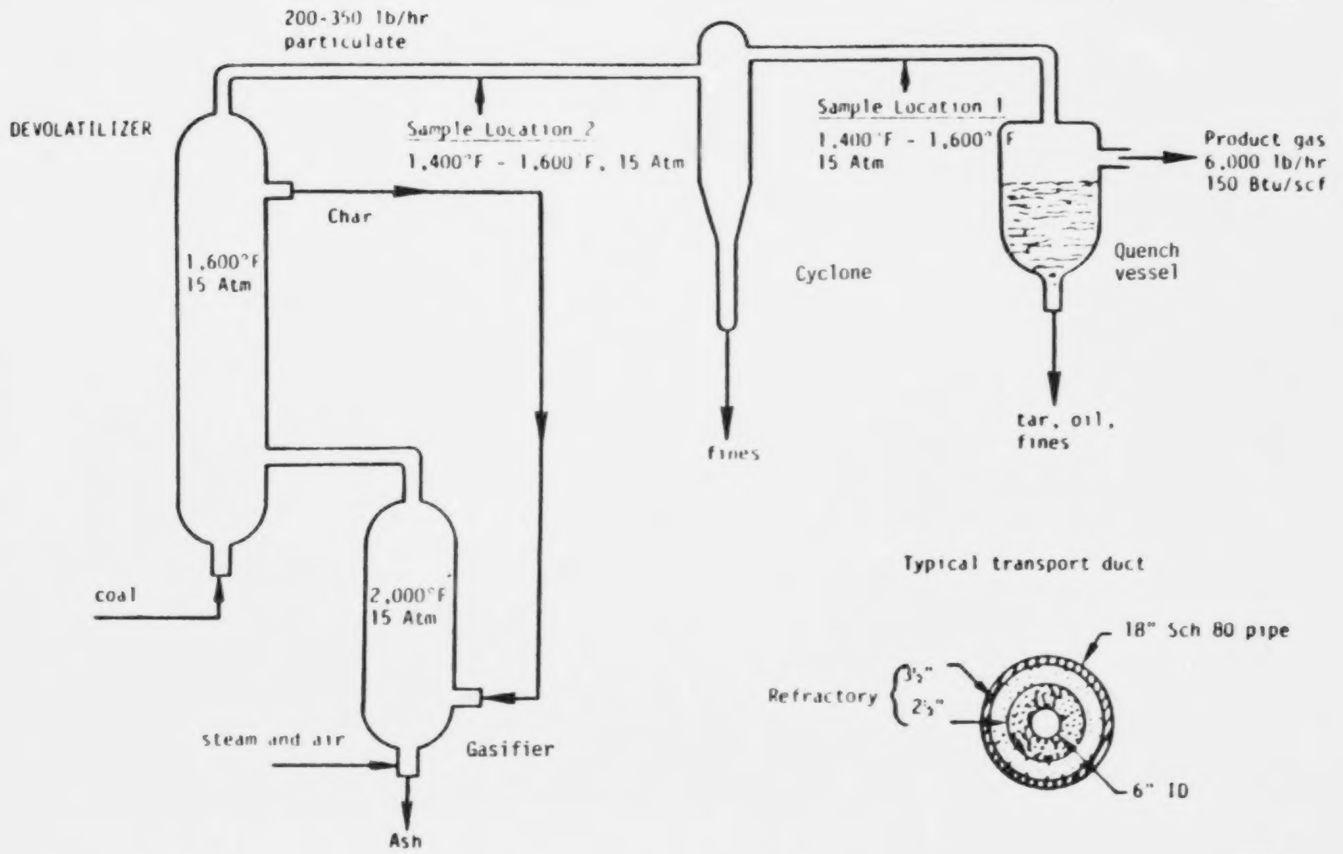
200-350 lb/hr
particulate
DEVOLATILIZER
Sample Location 2
1,400°F - 1,600°F, 15 Atm
Sample Location 1
1,400°F - 1,600°F
15 Atm
Product gas
6,000 lb/hr
150 Btu/scf
Char
1,600°F
15 Atm
Cyclone
Quench
vessel
tar, oil,
fines
fines
coal
2,000°F
15 Atm
steam and air
Gasifier
Ash
Typical transport duct
18" Sch 80 pipe
Refractory
3½"
2½"
6" ID

Figure 10. Westinghouse fluidized bed gasification process.

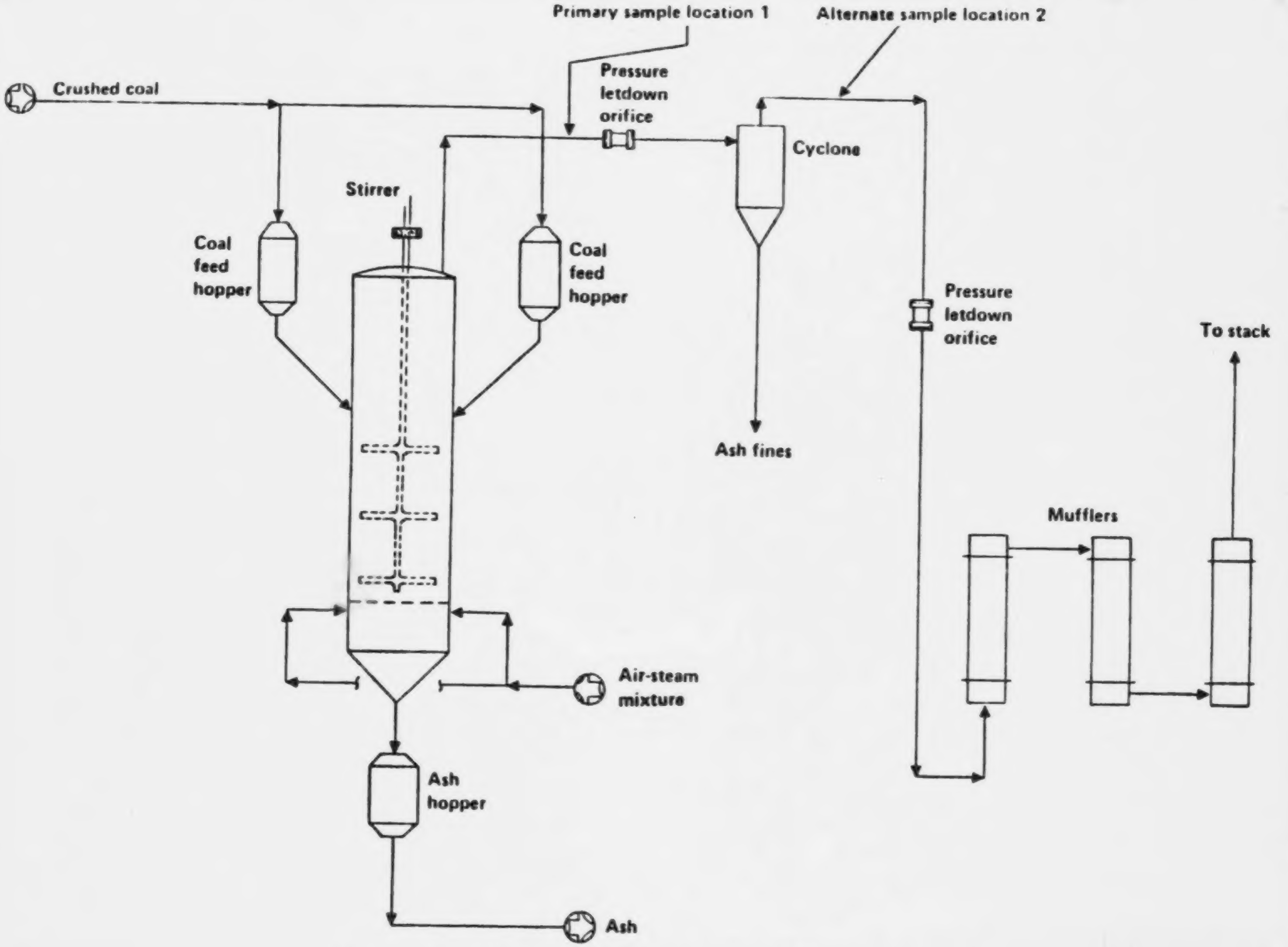
Primary sample location 1
Alternate sample location 2
Crushed coal
Pressure
letdown
orifice
Cyclone
Stirrer
Coal
feed
hopper
Coal
feed
hopper
Pressure
letdown
orifice
To stack
Ash fines
Mufflers
Air-steam
mixture
Ash
hopper
Ash

Figure 11. DOE MERC stirred bed gasification system.

TABLE 8. WESTINGHOUSE PDU GAS STREAM CHARACTERISTICS

Characteristic	Value
Temperature	1,400 ^{o}F to 1,600^{o}F
Pressure	225 psig
Stream velocity	22 to 23 fps
Approximate gas composition	CO:17.5%, CO_2:9%, H_2:13% H_2O:8%, CH_4:2.5%, N_2:50% (mole %)
Particulate loading	
Cyclone outlet	8 gr/acf to 20 gr/acf
Cyclone inlet	100 gr/acf

TABLE 9. MORGANTOWN STIRRED BED GAS STREAM CHARACTERISTICS

Characteristic	Value
Temperature	1200^{o}F nominal with excursions to 800^{o}F
Pressure	150 psig nominal, 300 psig maximum
Stream velocity	29 to 40 fps
Approximate gas composition (dry basis, mole percent)	CO: 16:2 percent, CO_2: 13.5 percent, H_2: 16.7 percent, H_2S: 0.5 percent, CH_4: 2.5 percent, N_2: 50 percent
Particulate loading:	
Gasifier outlet	10 gr/acf
Cyclone outlet	1 gr/acf
Tar and oil loading	
Gasifier outlet	17 gr/acf
Moisture content	10 to 20 percent

where a cyclone train was specified for catalyst removal upstream of a turbine gas expander.

Particle sampling in the MHD gas stream does not involve high pressures but does involve high temperatures. At the sampling location, temperatures were stated to be as high as 1370°C. The MHD exhaust also contains corrosive alkali seed material in addition to the gaseous and particulate products of coal combustion. These corrosives in combination with the extreme temperatures, present severe material selection problems for sampler design. One must be concerned about contaminating the sample collected through contact with the sampler probe itself. As MHD development continues, particle samplers will need to be adapted so that characteristics of particles in the MHD gas stream can be characterized.

Catalytic petroleum crackers operate at pressures of 2 to 3 atmospheres and exhaust temperatures of nominally 600°C. In the process the hot, high-pressure gas stream containing particles of catalyst is passed through a turbine device known as an expander. This step removes a portion of the energy in the gas stream and contributes in large part to making the process cost effective.

Catalyst contained in the gas stream is removed using several stages of cyclone separation. This removal recovers some of the catalyst material and is needed to protect the expander from erosion. Since the catalyst particles are originally large (1000 m) those small particles that leave the cracker are generated by grinding action between each other. Thus, they tend toward a larger size fraction capable of removal by inertial separation. Even so, such separation requires good cyclone performance to ensure turbine expander life of greater than 2 years.

To obtain the needed level of performance up to four cyclones in series are used. To monitor performance of these cyclone stages and help in improving designs to lower the fraction of particles released to the atmosphere through the turbine expander it is desirable to be able to measure particle concentration in the HTHP region of the ducting. A small market would exist for inexpensive general purpose samplers capable of operation in this application. However, the incentive for the petroleum industry or for cyclone manufacturers to pay for development of such equipment is lacking.

SECTION 3

SURVEY OF FACILITIES AND EQUIPMENT

As a part of the SOA review task of the overall program, a survey of potential HTHP sampler users was conducted. The reasons for conducting this survey were several. In the paragraphs that follow, the objectives of the survey will be discussed along with a discussion of the general findings.

3.1 OBJECTIVES OF SURVEY

The formal telephone survey was conducted over a period of 1 year beginning in April 1976. This formal survey was followed by more informal inquiries which continued until the end of the program; however, only the formal portion is reported here.

The first objective of the survey was to contact facilities involved in advanced coal conversion technologies to learn the goals and objectives of each of these programs from the standpoint of process development. Included in the inquiries was a determination of the operational environmental conditions at the locations where particulate sampling would be likely to occur. This included information such as the range of temperatures, pressures, and velocities that could be encountered.

Another objective was to determine what need, if any, currently existed or would exist in the future for particulate sampling within the process stream. In addition, information was sought as to whether particulate sampling would (1) be a necessity in terms of a tool for process development or (2) a means of acquiring additional information for determining process mass and energy balances. Inquiries relating to ground rules that might be imposed for sampling at a particular facility were also made. This would relate to safety requirements such as explosion proofing, typical constraints, and allowable shutdown periods for operation of the facility. Questions were posed as to whether any special problems such as sampler plugging and corrosive and/or erosive conditions that might inhibit sampling attempts.

Another objective was to determine what types of sampling had been done to date, the results that were obtained, and where the sampling equipment was obtained. Also, if sampling had actually be done (whether gaseous or particulate), what problems had occurred. If types of equipment were suggested or identified, inquiries were made to the manufacturers to determine their applicability to HTHP sampling conditions. General inquiries to experts in the field of coal conversion instrumentation were also made.

Finally, inquiries were made to identify process developers that might have an immediate sampling need which could be filled as part of the EPA program. This included inquiries to coal gasification developers who might have provided the opportunity to sample at their facility.

3.2 GENERAL FINDINGS OF THE SURVEY

Several members of the Acurex staff were involved in conducting the survey. The complete survey results are too extensive to cover in detail. Each phone contact was documented with a typewritten telephone memorandum and filed chronologically. These memorandum fill four 1-inch loose leaf notebooks. To condense this voluminous quantity of material, Table 10 was constructed. In this table, an effort was made to summarize some of the high points of the information that was collected. In addition to Table 10, a list of contacts was established that includes some persons that were not contacted directly during the survey, but who contacted us or attended conferences related to HTHP technology. This list is presented in Appendix A.

Without going into details of specific contacts, the results of the survey can be summarized as follows.

At the time of the survey, very few of the process developers were aware of the EPA-sponsored HTHP program that is the subject of this report. Most became interested, however, once the purpose of the program was explained. Many asked to receive additional information regarding the equipment that was under development.

During the period of the survey, most of the process developers were in the early stages of shaking down their facilities and bringing the systems up to desired performance specifications. In most cases, the processes had not been developed to the point where final stage cleanup devices had been brought online. In a few cases, the process was currently operating on a substitute fuel such as fuel oil or natural gas to test various operational characteristics of the process. In these specific instances, there was little immediate interest in performing in-process HTHP sampling. They did express the desire to perform particulate sampling at some point in the future once the final cleanup devices had been installed. This would naturally become a necessity to evaluate the performance of the final cleanup systems before the turbine stage could be installed.

Evaluation of final cleanup devices was the primary use to which HTHP sampling would be put as expressed by the developers. The second most often cited need for HTHP sampling was stated to be to establish mass and energy balances within the process. Where the latter need was expressed, HTHP sampling was of more near term importance to the process developer. For example, the viability of some processes depends upon the return of particulate captured in the first and second stage process cyclones to the reactor bed.

Naturally, one means of establishing quantities of particulate leaving and returning to the reactor is by virtue of particulate sampling. A few process developers even expressed the desire to be able to sample particulate

TABLE 10. SURVEY OF COAL CONVERSION DEMONSTRATION FACILITIES AND SAMPLING EQUIPMENT

Date	Facility	Person Contacted	Subject	Summary
4/12/76	A. D. Little	P. Levins	EPA in-house sampling task	P. Levins will discuss the ADL role in the EPA in-house sampling program during a visit to RTP during week ending 5/16. Suggested a mutual visit.
4/12/76	ERDA	G. Manning	Telecon to get acquainted with George Manning of Advanced Power Group of ERDA	Manning interested in capability of sampling inlet conditions to turbines. Would like to be invited to second sampling session at Exxon. Will assist Acurex in getting into a gasifier plant.
4/16/76	Radian	Gene Cavanaugh	Low Btu Gasification EA	Radian has not gotten into their EA to any great extent. Favors open communication.
4/20/76	Exxon/Linden	R. Hoke	Exxon pretest survey	Pretest survey to be held at end of May. Facility available for sampling on December 1976.
4/22/76	ERDA	F. Crim G. Manning	HTHP Sampling (Acurex-funded)	Interested in continuous mass monitor in HTHP environment for process control to protect turbine
4/23/76	ERDA	M. Sluyter	HTHP Sampling (Acurex-funded)	Involved in MHD development. Must sell uniqueness of HTHP sampler over conventional sampler. Suggest visit to John Dix, Coal MHD program at UTSI. Suggests we keep Sluyter informed of HTHP progress.
4/27/76	Mitre	J. Shang	Combustion Power Granular Bed Filter program	Chairman of Panel reviewing Combustion Power GBF program. Interested in hot particle collection. Indicates other possible sampling sites (FBC program at Rivesville, West Virginia, and PFBC program at Argonne).
4/30/76	EPRI	M. Gluckman	Telecon to identify potential coal sampling sites	"No operational full scale gasifier in the U.S.A. at present." Gave list of small scale gasification plants and contacts.
4/30/76	Gilbert Associates	W. Szwab	ERDA Sampling Survey	Briefed him on HTHP program

(continued)

TABLE 10. (continued)

Date	Facility	Person Contacted	Subject	Summary
4/30/76	ERDA	T. K. Lau	Instrumentation for gasifiers and FBC's	Brief discussion of HTHP program
4/30/76	Radian	G. Cavanaugh	Followup on Cavanaugh's visit to RTP	Radian's Low Btu/EA still in planning stage. Discussed manner by which Acurex HTHP-TLE will interact with Radian's EA Radian experts to work with Lurgi plant in Yugoslavia.
5/3/76	ERDA	G. Weth	Recent progress related to HTHP work	Curtiss Wright and GE have turbine cleanup contracts -- Leeds and Northrup and Spectron are developing particulate instrumentation
5/3/76	EPA/North Carolina	W. Kuykendal	More info on Weth call	Bill gave some background on L & N and Spectron contracts
5/4/76	Westinghouse/ Waltz Mill	D. Keairns	Westinghouse gasifier facility at Waltz Mill	Operates at 225 psi and 1600°F to 2100°F. They are interested in sampling for gases and particulates
5/5/76	Westinghouse/ Waltz Mill	D. Archer	Westinghouse gasifier	Interested in sampling upstream of cyclone -- gave physical data -- will relay needs to ERDA
5/5/76	Radian	G. Cavanaugh	Radian's ERDA contract to make gasifier measurements	Got organization roles straight -- any future work should be coordinated with Rhodes and Kuykendal
5/6/76	Westinghouse Research Labs	Brian Lancaster	Status of gasification plant and sampling	They will be using a simple approach but want to consider Acurex technology
5/11/76	Radian	F. Mesich	Radian's ERDA and EPA contracts	Have interest in SASS train -- general info on ERDA activities
5/12/76	ERDA/Pittsburgh	D. Nakles	Inquiry about HTHP program	He was interested in finding out more about HTHP work
5/12/76	Spectron	M. Farmer	Laser-doppler velocimeter	Description of apparatus -- will be testing at Argonne FBC

(continued)

TABLE 10. (continued)

Date	Facility	Person Contacted	Subject	Summary
5/13/76	Leeds & Northrup	S. Katy	Laser device to measure particulate size distribution	General info on program -- suggested a call to Dr. Muly
5/14/76	Westinghouse/ Waltz Hill	D. Archer	Arrangements for trip	ERDA not very interested in joint program with EPA
5/19/76	GE Schenectedy	J. Peterson	ERDA hot gas cleanup contract	Will be testing at Exxon -- interested in sampling during tests -- suggested we talk to GE gasifier people
5/20/76	Curtiss-Wright	C. E. Irion	ERDA hot gas cleanup contract	Will be using CW PFBC, 6 atm, 1350°F to 1700°F, using Rover turbine -- interested in HTHP work
5/21/76	Leeds & Northrup	Dr. Muly	L & N ERDA contract to measure particulates	Will be testing at Argonne Labs -- can measure to 1μ size over a 2-foot path -- testing to begin in 8 to 9 months
5/27/76	Caltech	R. Flagin	Discuss Academic contacts	Suggested I call Howard (MIT), Gavoles (Caltech), England (JPL) and Teixeira (EPRI)
5/28/76	MIT	J. Howard	Discussion of MIT coal gasification work	Has been doing sampling using water injection -- plans to sample at 100 atm - suggested call to J. Biordi at ERDA, Pittsburgh
5/28/76	ERDA/Pittsburgh	J. Biordi	Probe sampling reports	They will send reports -- interested in Acurex flame sampling work
6/1/76	Univ. of North Dakota	Gene Baker	Project Liquite	General info on operating conditions 2500 psi, 950°F high grade solid fuel
6/1/76	EPRI	Don Teixeria	HTHP sampling problems	Suggested we call KVB or UCB
6/1/76	Caltech	George Gavales	Cool Pyrolysis Research	Doing sampling at one atm. Using gas chromatograph and spectograph.
6/1/76	Exxon Miniplant	M. Nutkis	Field sampling	Discussed details of dimensions and valves required

(continued)

TABLE 10. (continued)

Date	Facility	Person Contacted	Subject	Summary
6/4/76	JPL	E. England	Sampling data	Flame and Combustion, and Coal-Fired atm furnace sampling. Problems with fuel rich regions, NO reduced by C.
6/4/76	G. E. Schenectedy	P. Palmer, Project Engineer, Power Cycles and fuel	GE/EPRI Gasifier Project: GE-GASD	Lurgi type gasifier operational, 170 psia 1100°F, eventually 350 psia and 300°F with spray part. Add-on. Receptive to sampling-ports available at top of bed.
6/8/76	Pitsburgh & Midway	J. Piatt, Tech. info officer	PAMCO's fuel processing plant to process low sulfur solid fuel from coal and liquid feedstock	50 to 100 ton/day plant operating at 1500 psig and 850°F. Have sampled using refinery techniques (grab).
6/8/76	Acurex/EPA	N. Jaffe	Additional info on PAMCO fuel processing plant from H. Fisher, ERDA	Obtained Project Report and Fisher suggested contact J. Ward, PAMCO regarding sampling techniques
6/10/76	PAMCO	J. Ward, Plant Mgr.	Obtain sampling information for PAMCO's solvent Refined Coal Pilot Plant	Sampling for process control, sample gases passed thru gas chromotograph. Liquids filtered and fractional distillation performed; interim reports forwarded.
6/14/76	Stone & Webster Boston, MA	R. C. Stone	Curtis Wright Fluidized Bed Combustor Gas-Turbine Project	Curtis to build small g.t. rig to test blade material also conceptual design (1977) for 500 MW utility.
6/17/76	Texaco Res. Lab., Montebello, CA	Dr. Slinger	Pilot Scale Gasifiers at Montebello	No technical information published on gasifiers pressure and temperature as high as 2500 psia and 3000°F
	Texaco Develop. Corp. New York, NY	J. Moranque, V.P.		
6/17/76	Bureau of Mines, Morgantown, W.VA	R. Casmer, Hardware Eng.	BuMines gasifier	ERDA funded. 100 to 300 psig, 1100°F to 1300°F. Sampling with Lear Siegler and Bendix gas chromatograph.
6/17/76	Atomics International Div/Rockwell International Corp.	A. Kohl	Pilot Coal Gasification Plant at Norwalk Harbor Station, CO, Light & Power Company	Project stopped but smaller scale plant to be built at Rockwell test facility, Canoga Park, CA in preliminary design

(continued)

TABLE 10. (continued)

Date	Facility	Person Contacted	Subject	Summary
6/18/76	U.S. Bureau of Mines, Pittsburgh, PA	H. Chambers	Hydrane Gasification Plant	One stage process operating at 1000 psi and 900°C. Gas chromatograph of H_2, CH_4, CO, CO_2, C_2H_6, H_2O, H_2S, C_3H_8. Problems with particulate plugging report to be sent.
6/18/76	Fluor Eng., Los Angeles, CA	J. M. Moe	Texas Eastern Transmission Co. Coal Gasification years plant at Four Corners, NM	Design of 26 units at 500 psia, 700°F completed. Construction delayed 2 to 3 due to funding problems.
6/18/76	Atomics International Div./Rockwell International Corp.	C. Trilling	Molten Salt Gasification Plant	Intermittent operating experience with pilot plant 1 atm at 1700°F to 1900°F under contract to ERDA to build 5-ton/hr pilot plant (startup late 1977) at 1 to 20 atm and 1700°F to 1900°F
6/22/76	Argonne Labs	Dr. N. O'Fallon	Article "Monitoring Coal Energy Processes" in Processes" in Industrial Research, June 1976, co-authored by O'Fallon	To send copy of Argonne report to be completed in next few months
6/29/76	Radian Corp.	G. Cavanaugh	Six Lurgi gasifiers installed in Yugoslavia	Plant operates at 300 to 350 psi, interested in sampling (grab)
7/2/76	U.S. Bureau of Mines Synthane Coal Gasification Plant	Dr. J. Strakey	Synthane Coal Gasification Plant	Plant to produce high-Btu pipeline gas from coal. Gas cleanup by cyclones followed by scrubbers sampling with gas chromatograph downstream of scrubbers. No interest in outside help with sampling.
7/2/76	Battelle	W. Corder	Ask Agglomeration Gasifier	Plant has two fluidized beds; one gasifier and one combustor at 100 psi and 1600°F. Interested in both gaseous and particulate sampling.
7/2/76	USBM Synthane Plant	Dr. J. Starkey	Interest in sampling program and fact finding	Had not started to gasify coal. Doing their own sampling.

(continued)

TABLE 10. (continued)

Date	Facility	Person Contacted	Subject	Summary
7/2/76	Battelle ash agglomeration gasifier	W. Corder	Interest in sampling program and fact finding	Will be fully operational in about 2 months -- 1800^{o}F and 100 psi have been doing gas sampling themselves; will be interested in sampling ahead of turbine
7/2/76	Morgantown Energy Research Center	A. Liberatore	Interest in sampling program and fact finding	Are currently sampling at four locations for particulate and gases -- having problems with tar condensates
7/2/76	Curtis-Wright	E. Garruto	Guidance in sampling at CW	Receptive to an EPA-funded sampling program (95 psia, 1700^{o}F)
7/6/76	CO_2 Acceptor Plant	T. Holen	Interest in sampling program and fact finding	Have done some sampling at 150 psi and 1500^{o}F particulate clogging problems -- interest in gasifier overhead sampling
7/6/76	Bi-gas	D. Hull	Interest in sampling program and fact finding	Sampling for gases, 1500 psi, 1700^{o}F. Interested in method of measuring char.
7/9/76	Radian	G. Cavenaugh	Plans to sample at CO_2 acceptor	Under ERDA contract -- not sure if they will be sampling at CO_2 acceptor
7/9/76	McDowell-Wellman	R. Wellman	Wellman-Galusha gasifiers	Identified three operational units in Penn/Ohio region
7/23/76	GE Gasifier Schenectady	J. Wang	HTHP Sampling	Sampling for condensed tars, 10 to 20 atm, 300^{o}F to 600^{o}F
7/26/76	U.C. Berkeley	B. Macknick	ERDA-sponsored tar characterization program	Discussed funded program to determine vapor pressure and condensation temperatures of CG tars
8/3/79	CO_2 Acceptor	T. Holen	Sampling interest and sampling plans by Radian	Radian will make trace element mass balances for process streams under ERDA contract
8/3/76	Energy Research Center Grand Forks	R. Ellman	Interest in sampling program	GFERC has interest in particulate sizing on the four-stage condensor
8/5/76	Curtiss-Wright	D. Weller	Sampling interest	Desire opacity, NO_x, SO_x, particulate, etc., measurements

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TABLE 10. (continued)

Date	Facility	Person Contacted	Subject	Summary
8/26/76	Energy Research Center Grand Forks	R. Ellman	GFERC operational status and Ellman contact with ERDA	ERDA has not responded to Ellman yet. GFERC currently operating at 200 psi. Current sampling needs are low.
8/30/76	U.C. Berkeley	B. Macknick	Tar characterization study for ERDA	Methods of tar collection, storage, and handling summarized. Possible consulting arrangement discussed.
9/3/76	Environmental Systems Corp.	B. Nuspliger	PILLS devices	General discussion of product line
9/9/76	Stearns-Rodger	B. Scheck	ERDA, FBC award	Followup call to determine HTHP sampling needs
9/10/76	Riley-Stoker	S. Johnson	Tar collection techniques	Tars form fine mists they used millipore filters to remove them
9/10/76	GFERC	R. Ellman	Interest in HTHP	They have run for 3 to 4 hours on lignite at 200 psi -- will go to 400 psi in October
9/15/76	Bi-gas	D. Hull	Progress update on Bi-gas	Still not operating on coal; expects to be coal fired in late October
9/20/76	GFERC	R. Ellman	Sampling in GFERC	Ellman to call Grua for impact to his decision
9/20/76	CO_2 Acceptor	T. Holen	Status of CO_2 acceptor	Discussed sampling operational problems tar problems
9/20/76	Westinghouse/Waltz Mill Gasifier	Suresh Tendulkar	Tar collection at Waltz Mill	Tars are in the 8 to 10 percent of coal feed range -- ESP's are effective in removing mist from CG lines; property data lacking
9/23/76	Sandia Corp	D. Hartley	Sandia ERDA Project	Funded to provide diagnostic equipment assessment for coal-fired combustion processes
9/24/76	Riley-Stoker	B. Lisauskas	Tar properties	Separate by size using cascade impactors; molecular wts = 300 to 800 lb/lb-mole; also gave gas properties

(continued)

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TABLE 10. (continued)

Date	Facility	Person Contacted	Subject	Summary
10/4/76	Energy Research Center Morgantown	Dr. M. F. Scharff	Instrumentation development for Morgantown FBC	In-bed measurements of specie, flow, and thermodynamic data are critical to FBC development
10/6/76	Energy Research Center Grand Folks	R. Ellman	Ellman discussions with with Grua re Acurex sampling	Not currently feasible due to intermittent operation
10/6/76	ERDA Oak Ridge	A. Sidparo	ERDA funding of FBC unit to be built at Univ. of West Virginia by Reynolds, Smith, and Hills Co.	Unit is 500 psi, 700° to 1000° steam produced by 10 to 90 tons/hr coal. Cleanup system design is preliminary.
10/7/79	U.C. Berkeley	B. Macknick	Support of gasifier tar studies	Macknick to provide state-of-art exchange
10/12/76	ERDA	C. Grua	Acurex sampling at GFERC	Inquired as to availability by GFERC site by sampling
10/26/76	Energy Research Center Grand Forks	R. Ellman	C. Grua's comments to N. Jaffe re HTHP sampling with EPA	ERDA/EPA expected to be cooperative. Acurex should submit a WR which addresses: (1) Design and (2) sampling locations of HTHP
10/28/76	Energy Research Center Grand Forks	R. Ellman	Sampling at GRERC	Sampling spec: 200 psi, producer gas 300°F to 400°F, no evidence of tar (may be a fine mist). Relocate sampling equipment for convenience.
11/22/76	GFERC	R. Ellman	Sampling work recommendation	Agreed that it did not make sense to submit a work recommendation at this time
11/30/76	GFERC	R. Ellman	Tar problems at GFERC	Compared the effectiveness of scrubber and side stream sampler. Will be making another run at 200 psi, if successful will run at 400 psi.

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TABLE 10. (continued)

Date	Facility	Person Contacted	Subject	Summary
12/15/76	Molten Salt Gasification Plant	C. Trilling	Tar problems	They don't think they have a tar problem at operating temps (1700°F to 1900°F) yet they have seen some evidence of tar accumulation. He was interested in particulate plugging problems.
12/22/76	GFERC	D. Mioduzewski	HTHP update	Particularly interested in production rates of water, tars, particulates, and gases. Will provide us with tar samples.
12/27/76	Battelle, Ash Agglomeration Gasifier	W. Corder	Tar problems	Haven't started operating gasification leg yet. Not sure if a tar problem exists.
12/28/76	Bi-Gas	D. Hull	Tar problems	Have just started operating on coal. No tar problems observed yet. Feels that high temps will crack tars (1700°F/3200°F reactor temperatures).
12/29/76	Jaycor	M. Scharff	Instrument developments	Suggested we contact GE on impact probe and Magna Co. on corosometer
12/30/79	Westinghouse/Waltz Mill Gasifier	S. Tendulkur	Tar problems	Currently accumulating tars in quench phase. They are interested in being able to sample for tars -- especially after combustor/gasifier leg is operational.
1/4/77	GE (Schenectedy)	Dr. Ralph Wood Dr. Steven Brzozowski	Development of a Particle Impact Probe	Can measure velocity and momentum of particles down to 635 micrometers. In erosion field 15,000 to 20,000 impacts per second.
1/13/77	McDowell-Wellman	Wallace Hamilton	Wellman-Galusha Gasifiers	Attempts to sample producer gas derived from bituminous coal in Wellman-Galusha gasifiers have failed due to tar accumulation
1/18/77	Magna Corp, Santa Fe Springs, CA	Jerry Lambert	Corosometer	This instrument measures corrosion and erosion in gas and liquid streams. Widely used; including coal gasification units.

(continued)

TABLE 10. (continued)

Date	Facility	Person Contacted	Subject	Summary
1/18/77	Riley-Stoker	Tom Walsh	Information on their gasifier	The unit is a full scale Riley-Morgan design. 7500 lbs/hr with air and 1300 lb/hr with oxygen. Cleanup train will be required to clean up tars.
1/19/77	Morgantown Energy Research Center	C. C. Shale	Sampling of Stirred Bed Producer Gasifier	Sampling attempts thus far have been unsuccessful due to the inability of their system to maintain high enough temperature to avoid tar plugging. Biggest problem is between cyclone and two-stage venturi scrubber.
1/20/77	Holstein Army Arsenal Gasifier, Kingsport, Tennessee	Clarence Berryman	Gasifier Sampling	They own a Chapman (Wilputte) gasifier for producing low Btu gas. They do in-house gas sampling periodically. Unit has been operating for 25 years.
1/20/77	American Natural Gas	Dr. Fred Jones	American Natural Gas Test Program at Riley-Stoker	Evaluating the performance of lignite coal. Sampling will be done at 600°F. Radian involved. American Natural Gas is also involved in an FBC test at Foster-Wheeler.
1/21/77	Stearns-Rodgers (Denver)	Bob Scheck	HTHP Sampling and Gas Cleaning	Stearns-Rodgers is doing E&A at Argonne Labs PFBC. He wants to be kept informed on HTHP sampling and gas cleaning.
1/25/77	Fluidynamic Devices, Limited	Richard Wirth	Sampling at Exxon	A special probe has been developed to measure accurately low velocity flows (8 fps). Operates on fluidic amplification principle.
1/26/77	U.C. Davis	Bob Williams	Blue Diamond Gasifier	Their walnut shell gasifier produces complex tar/matter. Other fuels are corn cobs, tree pruning and wood chips. Efforts to sample by condensing and filtering the tars have been unsuccessful.
1/26/77	Pittsburg and Midway Coal Co.	Dr. Perussel	Sampling needs at SRC plant	They currently use a powdered coal slurry and are in the process of converting to coal liquifaction operation. Perussel indicated they are "clean" from an EA standpoint since they have a closed system.

(continued)

TABLE 10. (concluded)

Date	Facility	Person Contacted	Subject	Summary
2/1/77	Hydrocarbon Research	Harold Stotler	Sampling work on new EPA program	Evaluating fuel conversion cleanup. May volve sampling in about a year.
2/8/77	Grand Forks Research Center	Robert Ellman	Slagging Lurgi gasifier	A recent run at 400 psi resulted in slagging problems. Their sampling system is producing good results without tar plugging.
2/9/77	Synthane	Robert Lewis	Facility sampling plans	Early design stage for sampling system by Lummis Co. based on a lab scale system. Anticipate tar problems.
2/11/77	Morgantown Energy Research Center	Al Moore	Gasifier stream characteristics	Varying particulate loadings may make measurement difficult. Light oil separation from water may not be possible.
2/14/77	GE (Schenectedy)	Clem Thoennes Walt Jiles	HTHP samplings and hot filtration application	They are presently reviewing HTHP sampling capabilities. Current materials evaluation work is being performed at Exxon.
2/17/77	U.C. Berkeley	Brian Macknick	Concept evaluation on tar collection	Throttling was identified as a viable technique for condensing tars. A light oil/water separation technique was identified. Tar collection concepts were reviewed.
2/17/77	EPA	Bruce Henshel	Exxon PFBC Sampling plans	Cyclones will be used to evaluate the granular bed with three cut sizes below 1 µm. Particulate to be removed at 1600°F, 10 atm.
3/4/77	Bi-gas, Homer City, PA	D. E. Hull, *et al.*	Sampling requirements	Present interest in solid sampling process. Sampling activities will come later.
3/11/77	Stearns-Rodgers	R. Crawford, *et al.*	Sampling requirements	PFBC facility for Argonne will require sampling hardware yet to be developed.
3/14/77	Argonne Labs	J. Montagna	Sampling of Stearns-Rodgers PFBC unit	An RFP for bed and process sampling will be issued for the unit to be completed in 1980.
3/14/77	MERC	A. Moore	ERDA sampling of alkali metals in MERC 16" gasifier	Sampling to be performed downstream of scrubber and hot gas cleanup atomic emissions spectrometer to be used for sodium and potassium detection. Currently need a technique to separate particulate and tars.

and slag in the reactor itself, a task that is beyond the current state of the art.

Several process developers mentioned problems that they anticipated would occur or had actually occurred during sampling attempts. A very limited number of developers had attempted particulate sampling using very rudimentary devices such as fixed probes and single filter traps. At the time of the survey, no one had produced an operational particulate sampling system of the nature of that developed under this EPA-sponsored program. The results that had been obtained by the process developers in their attempts to sample were not clearly definitive. Most of those who attempted sampling had encountered problems associated with particulate plugging, tar condensation (in gasifiers), and acid gas condensation. Some of the gasifier developers who had not yet attempted in-process sampling at the time did express deep concern about the problems that they anticipated once sampling was a requirement to continue process development. Most of their concern centered about problems relating to tar condensation, probe material survival, and safety considerations. Many mentioned problems of tar condensation that had occurred within the process itself during normal operation.

Finally, the conversation often got to the point where the process developers asked the approximate cost of developing a sampling system that would meet the needs of their particular process. The response to the high cost of HTHP probe development was generally that the process developer would not be able to provide the required funds. In nearly all cases the developer responded that they would need to request the funds from the funding agency. That is to say, the cost of sampler development had not been anticipated or included in the development funds that were currently available.

SECTION 4

APPROACHES TO HTHP SAMPLING

In this section we will consider some of the general aspects of selecting an HTHP sampling system and its requisite components. These discussions are based upon information surveyed from the literature from direct contact with manufacturers and other users and from experience gained from direct sampling activities during this program.

4.1 CONTINUOUS VERSUS NONCONTINUOUS SAMPLING

In selecting a sampling system for application to a high-temperature, high-pressure technology the first question that must be posed is that of the exact purpose and intent for which the sampler is to be used. At the present time all of the coal conversion technologies discussed in this report (fluidized bed combustion, coal gasification, and MHD) are in a state of development. Many different problems are yet to be resolved; however, one very significant problem that has come to the forefront in all of these technologies is that of particulate control.

In the evaluation of particulate collection devices for coal conversion systems it is necessary to take samples both upstream and downstream of the collection system to establish its performance. The information most frequently desired is that of total particulate concentration, particulate size distribution, and particulate matter chemical composition.

Two general classes of particulate sampling systems are available, those that operate on a continuous basis (more commonly referred to as monitoring devices) and those that operate noncontinuously or intermittently. Monitoring devices generally do not possess the capability of physically extracting a sample of the particulate for chemical analysis. Continuous devices tend to be nonintrusive or _in situ_ devices while noncontinous sampling systems tend to be intrusive or extractive devices. That is, they are inserted into the flow and physically remove a sample. A good example of a noncontinuous extractive sampling system would be the one developed under this program for use at the PFBC at Exxon. Similarly, a good example of a continuous _in situ_ monitoring device would be an optical sampler based upon laser optical scattering.

4.1.1 _Optical Systems_

Several optical systems are currently under development by both the EPA and DOE. At this point in their development, these optical devices have not been useful as instruments for evaluating the performance of particulate collection systems and other portions of advanced coal conversion systems, but may adequately serve as monitors to determine system upsets and perform estimates of particulate loading levels and size distribution. A brief survey was performed under this contract to evaluate the availability of SOA particulate monitoring systems for application to HTHP systems. The purpose of the survey was, in part, to determine if any of these systems could have been used in conjunction with the PFBC probe during the Exxon tests to compare the performance of both. No such attempt was made however. The results of this survey are presented in Appendix B. A more detailed discussion of optical systems for use in HTHP stream can be found in Reference 3.

For particle size distribution determinations, extractive samplers separate the particulate using inertial devices (cyclones or impactors) that directly correlate to the so-called aerodynamic diameter of the particles. Size classification by aerodynamic diameter is particularly useful in evaluating particulate collection devices that depend upon aerodynamic impaction behavior as their means of particle capture. Consequently, correlations between the operation of these particle collection devices and particulate sampling by an extractive sampler are direct.

On the other hand, optical systems tend to measure an effective optical diameter. This diameter depends upon the optical light scattering properties and the shape of the particle. The particle diameter that is measured is not aerodynamic, and some assumptions must be made to compare optical aerodynamic data (Reference 16). Consequently, the usefulness of optical diameter as a means of determining particulate removal system performance is of less than optimum value.

Other problems must be resolved before optical systems will come into more general usage as monitoring devices. The operation of these systems often depends upon the use of one or more optical windows located on the side of the duct through which a laser beam and scattered light must pass. The successful operation of the device depends upon keeping the windows optically clean. This may be a significant problem over long operational periods with constant exposure to corrosive gases, slag, tars, and sticky particulate matter. The windows can be recessed from the duct wall and a purge stream applied as a possible means of keeping them clean.

Another problem associated with optical monitors operating in high-temperature streams is that the background radiation of the hot gases may interfere or tend to mask the optical signals received by the monitoring system. Also optical systems may be limited to use in situations with light particulate loadings. If more than a single particle occupies the measurement volume at one time the measurement is invalid. Also high dust loadings can attenuate the light beam and thus limit its use.

In summary, optical systems and extractive systems tend to be complimentary and not competitive measurement devices at present. The extractive approach allows physical and chemical analysis of the sample while optical monitors can provide process developers with on-line evaluation of system upsets and approximate particulate loadings and size distributions.

Future improvement of extractive and optical systems will determine whether the two approaches will tend to become more competitive as analytical aids in process component development.

4.2 APPROACHES TO EXTRACTIVE SAMPLING

This section discusses two topic areas: the design concepts appropriate to fixed position sampling and, secondly, various means of designing the pressure containment vessel for traversing probe applications.

4.2.1 Fixed Probe Concepts

The question of whether fixed or movable probes are used depends upon the application. For most R&D work the flowfield must be surveyed, consequently, traversing probes are required, whereas, fixed probes may be used where surveys are not required. Figure 12 shows two different fixed probe designs. Figure 12 illustrates a probe which is truly fixed into a port plate at one position in the duct. It will be shown that such an arrangement has limited application. Since the probe is continuously exposed to the duct environment it is subject to erosive and corrosive attack, and such failure of the probe may lead to damage of downstream components.

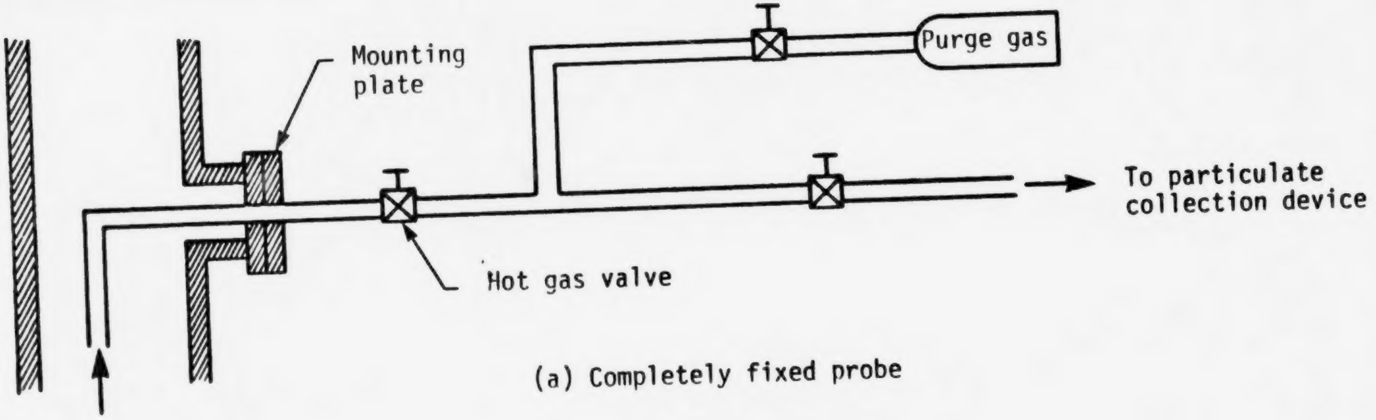
In addition, some means of keeping the probe free of particulate matter while not in use must be provided or probe clogging will occur. Two possible methods of accomplishing this include maintaining a constant external gas flow purge while the probe is not in operation. This requires large quantities of purge gas. A second approach is to turn the probe using a rotating seal, so that the probe is facing downstream while it is not in operation. An external valve must be provided to allow a sample to be withdrawn. After some period of probe use, the sample tube should be cleaned which would require shutdown of the process stream for probe removal. Consequently, the completely fixed probe, although simplest in concept, lacks practicality for particulate sampling. Is is more appropriate for gas sampling systems free of particles.

A retractable-fixed probe design is shown in Figure 12(b). In this design the probe is either entirely withdrawn from the duct or fixed at a single point in the duct when in use. Once inserted into the duct, positive sealing occurs. Purge gas is only required during probe insertion and extraction. When the probe is not in use and retracted, it is protected from the hot gases by the closure of a gate valve. This type of fixed probe is much more practical than a completely fixed version yet is somewhat less costly than a traversing system.

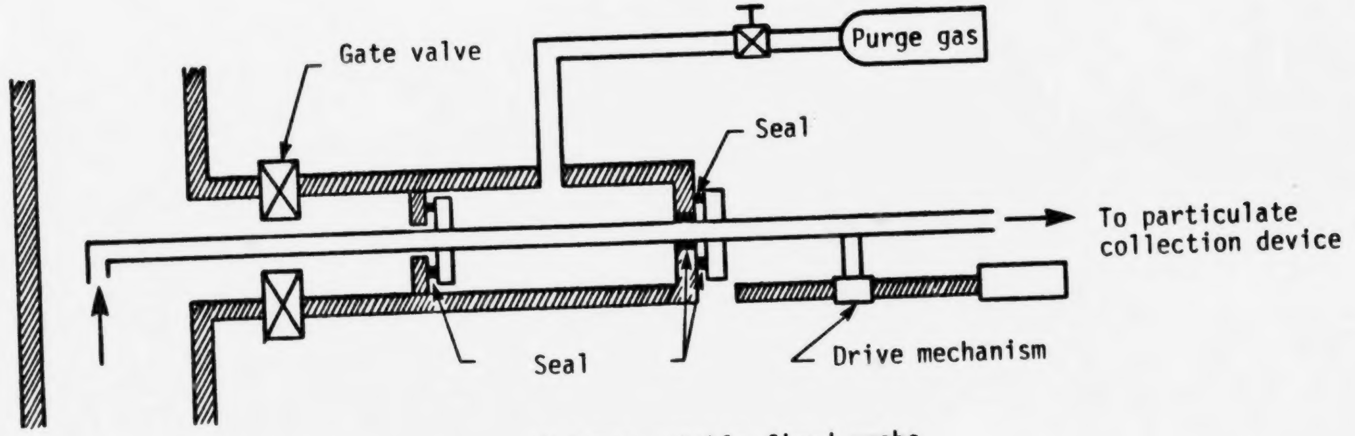
When it is necessary to survey the duct a traversing system must be used. Various approaches to a traversing design are considered in the following paragraphs.

4.2.2 Traversing Probe Concepts

Several approaches to traversing probe systems were considered during the course of this program. Three basic alternatives are shown in Figures 13,(a) (b), and (c).


Mounting plate
Purge gas
To particulate collection device
Hot gas valve

(a) Completely fixed probe


Gate valve
Purge gas
Seal
To particulate collection device
Seal
Drive mechanism

(b) Retractable-fixed probe

Figure 12. Fixed probe concepts.

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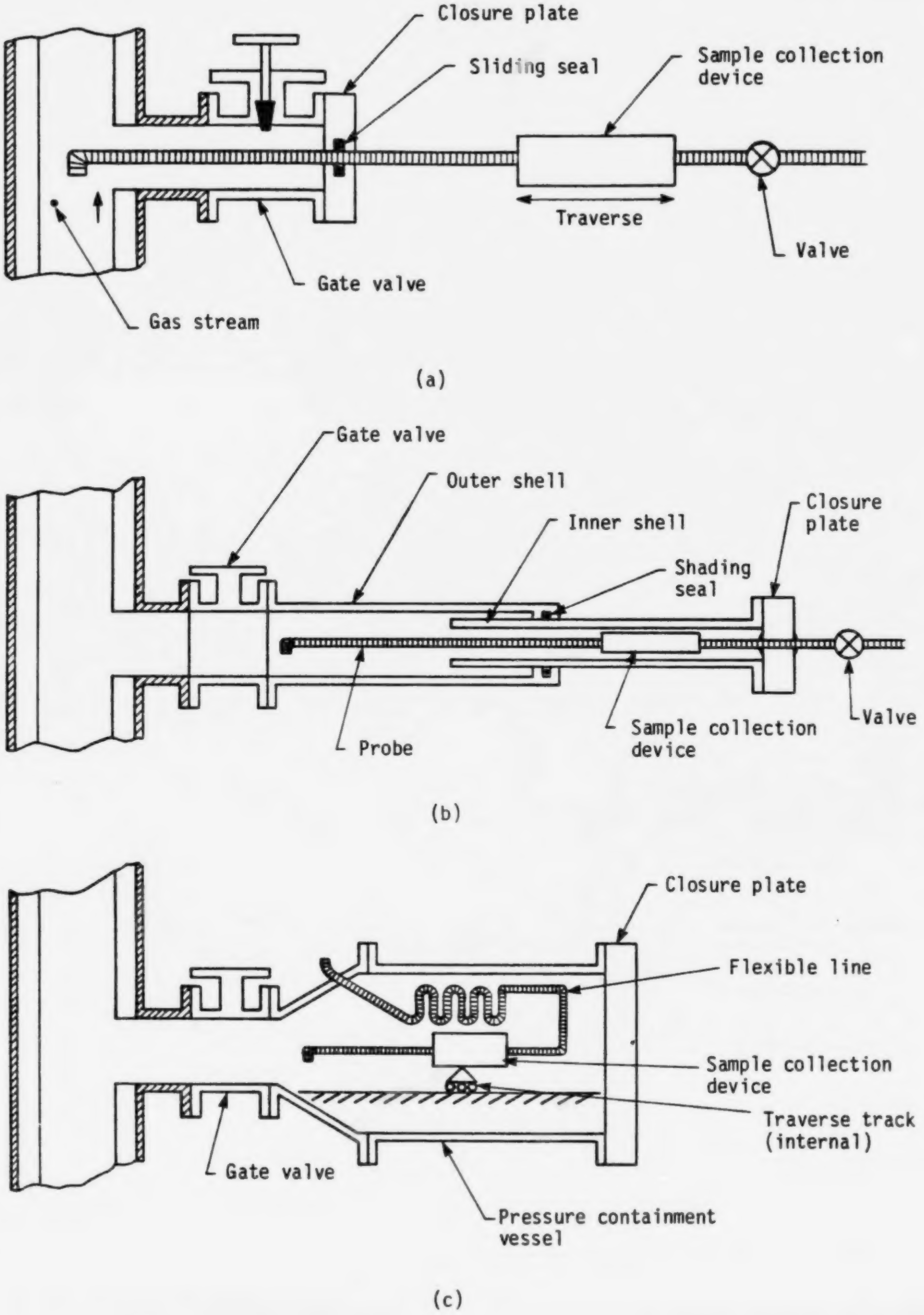
Closure plate
Sliding seal
Sample collection device
Traverse
Valve
Gate valve
Gas stream
(a)
Gate valve
Outer shell
Inner shell
Closure plate
Shading seal
Sample collection device
Valve
Probe
(b)
Closure plate
Flexible line
Sample collection device
Traverse track (internal)
Gate valve
Pressure containment vessel
(c)

Figure 13. Traversing probe.

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Sliding Seal

Figure 13(a) shows a traversing probe concept termed the sliding seal concept. The principal advantage of this approach is that the physical size and weight of the system are considerably reduced over that of other approaches considered. The main pressure housing can be made of minimum size. The sample probe tube must be rigid and straight to its juncture with the particle collection device to avoid particle deposition. This requires that the particle collection device slide with the probe. Having the particulate collector external permits easy access. The sample collection enclosure can be a pressure vessel itself or the walls of the sample collectors can act as the pressure vessel. Also, because the diameter of the sliding tube is likely to be small, the blowoff loads and the actuation requirements are minimized.

On the negative side, the sliding tube and seal are exposed to hot corrosive gases and particulate matter. Sealing surfaces can quickly degrade under such conditions leading to a system leak. Various means can be devised to protect the seal and metal surfaces such as purge systems and telescoping shields over the sample tube.

Concentric Tube

The traversing probe concept shown in Figure 13(b) is termed the concentric tube. In some respects it is similar to the sliding seal concept. In this configuration, the sample collection device is contained within the inner shell that acts as the pressure vessel. The sliding metal to seal surface is easier to maintain in that a wiper can be employed at the forward end of the inner tube to keep particulate and corrosive materials off of the outer surface of the inner shell. The concentric tube type of design was employed for the Exxon PFBC probe.

The principal disadvantages of this design are that the size and weight of the system tend to be large. Also, because of the relatively large diameter of the inner shell the blowoff loads generally will require the use of a hydraulic or motor driven traversing mechanism.

Total Enclosure

The third approach shown in Figure 13(c) is called the total enclosure concept. Here the pressure vessel is fixed and the sample tube and collection device move on a track within the vessel. No sliding seals are required and since no movable surface passes through the pressure vessel, there are no blowoff loads to overcome during traverse. Consequently, the traversing can be done manually using a chain drive or rack and pinion mechanism.

The major disadvantage of the third concept is that of size and weight. Since the vessel diameter is the largest of the three, the wall thickness and, therefore, overall weight may be the largest. In general, however, the total enclosure concept will be shorter than the concentric tube concept.

Table 11 summarizes the various advantages and disadvantages of each of the three concepts. Depending upon the particular application each of the three approaches may be most advantageous in a given circumstance. In general, however, the total enclosure approach seems to offer several attractive advantages.

4.2.3 Prototype Designs

To illustrate the application of each of the design concepts Figures 14, 15, and 16 are presented. These schematics show three different PFBC applications in which each of the respective design concepts was selected. All major system components are identified.

In addition, Figure 17 is presented to show a schematic of the total enclosure approach applied to a gasifier sampling system. Figure 18 shows a conceptual design of the same gasifier sampling system.

In summary, the total enclosure approach seems to offer the most flexibility in design concept. Internal components may be selected so that different sampler configurations can be achieved depending upon the application. For applications where sampling at a fixed location is permissible the semifixed concept offers the most practical approach.

4.3 PARTICULATE COLLECTION DEVICES

Several techniques are available for use in a sampling probe to collect and classify the particulate by size. These methods include cyclones, impactors, filters, impingers, diffusion batteries, and electrostatic precipitators (ESP). In designing a sampling system for use in a HTHP application, a selection of the most appropriate particulate collection device must be made. The discussion that follows is limited to the first three devices, in that they are most commonly used in conventional extractive samplers and can be adapted to use at HTHP conditions. This is not to say that the other devices mentioned or others not mentioned might not be appropriate in a given situation. For example, the ESP may provide the best means of collecting fine tar mist when sampling at a coal gasification facility. This is discussed further in Section 5.3. The reader is referred to Reference 9 for a discussion of particulate collection devices applied to HTHP sampling conditions.

4.3.1 Impactors, Cyclones, and Filters

As mentioned previously, three different types of particulate collection devices will be considered here as the most likely to be selected for near term HTHP sampling systems.

Impactors generally consist of multiple stages of plates, each containing a number of progressively smaller holes arranged in series for collection of particles by aerodynamic diameter. Each plate contains a number of holes through which the particulate laden stream flows in a jet-like manner. The gas jet impinges perpendicular upon the plate immediately downstream of the orifice plate. As the flow continues downstream, the holes in each stage are

TABLE 11. SUMMARY OF TRAVERSING PROBE CONCEPTS

Concept	Advantages	Disadvantages
Sliding seal	• Simple in concept • Light weight • Smallest envelope required for probe insertion/removal	• Seal is on probe; it sees the hot corrosive environment. Not practicable for high temperature environments, therefore seal must be protected and cooled.
Concentric tube	• Seal is on a cold clean surface • Sample collection device can be enclosed in pressure environment	• Heavy pressure chamber • Requires long envelope for probe extraction extraction and removal • Seal diameter relatively large -- blow-off loads significant requiring power assisted traverse (hydraulic, or electric drive)
Total enclosure	• No sliding seals required • No power assisted transverse required • Reasonable envelope required for probe insertion/removal	• Pressure chamber may be larger in diameter than concentric tube • Overall weight large

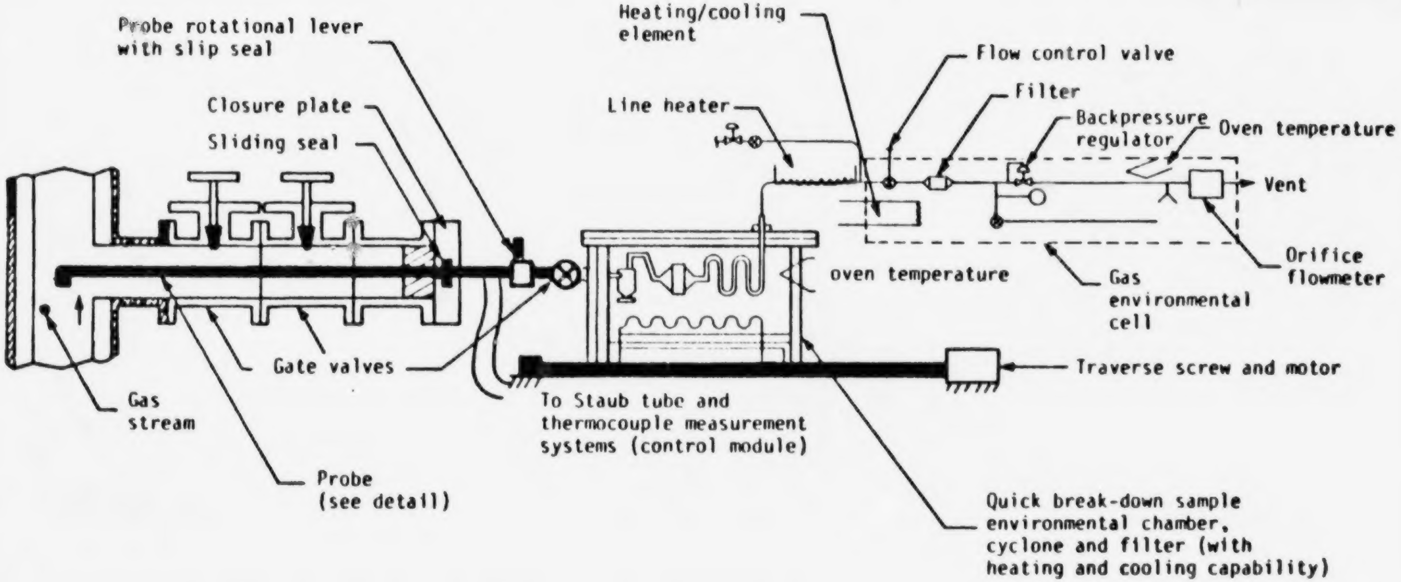
Probe rotational lever with slip seal
Closure plate
Sliding seal
Gate valves
Gas stream
Probe (see detail)
To Staub tube and thermocouple measurement systems (control module)
Heating/cooling element
Line heater
Flow control valve
Filter
Backpressure regulator
Oven temperature
Vent
Orifice flowmeter
oven temperature
Gas environmental cell
Traverse screw and motor
Quick break-down sample environmental chamber, cyclone and filter (with heating and cooling capability)

Figure 14. Schematic of sliding seal sampling system.

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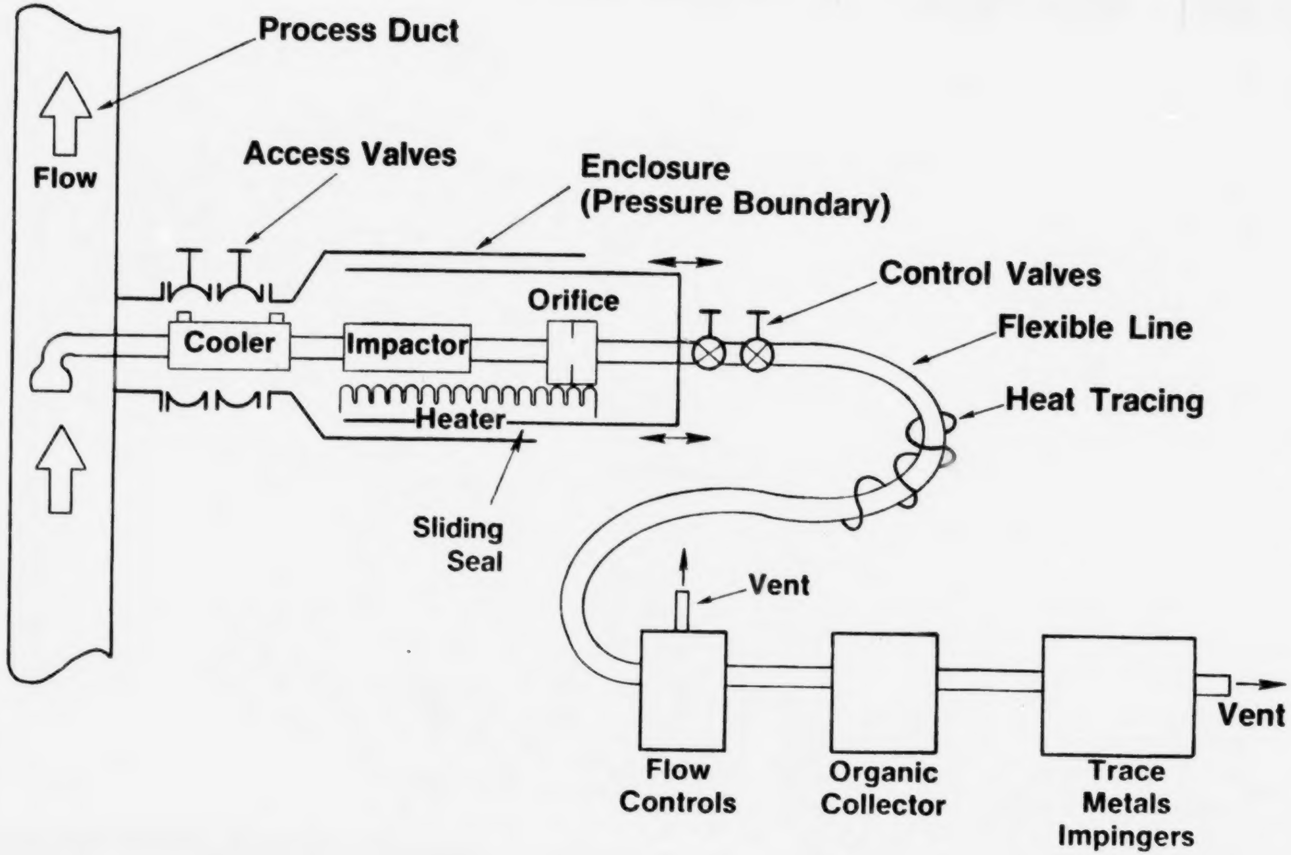
Process Duct
Flow
Access Valves
Enclosure
(Pressure Boundary)
Control Valves
Flexible Line
Orifice
Cooler
Impactor
Heater
Heat Tracing
Sliding
Seal
Vent
Vent
Flow
Controls
Organic
Collector
Trace
Metals
Impingers

Figure 15. Schematic of concentric tube sampling system.

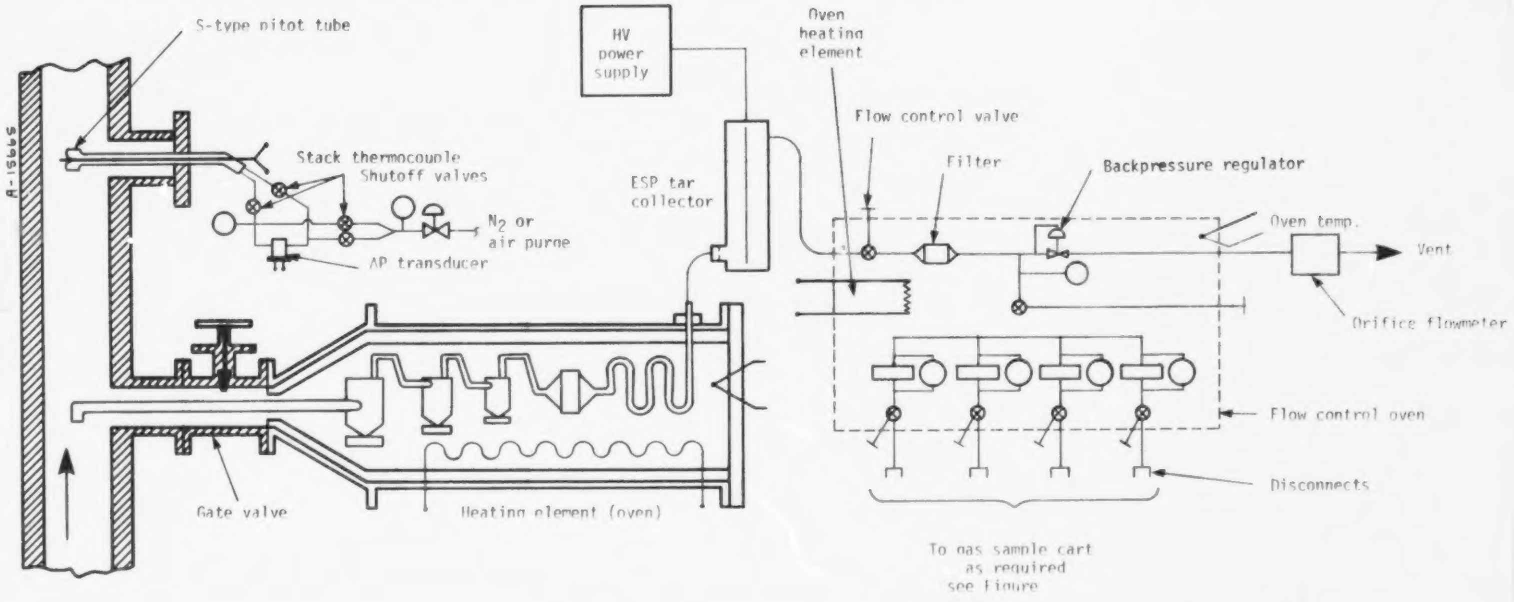
S-type pitot tube
Stack thermocouple
Shutoff valves
N_2 or air purge
ΔP transducer
Gate valve
Heating element (oven)
HV power supply
ESP tar collector
Oven heating element
Flow control valve
Filter
Backpressure regulator
Oven temp.
Vent
Orifice flowmeter
Flow control oven
Disconnects
To gas sample cart as required see Figure
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Figure 16. Schematic of total enclosure sampling system.

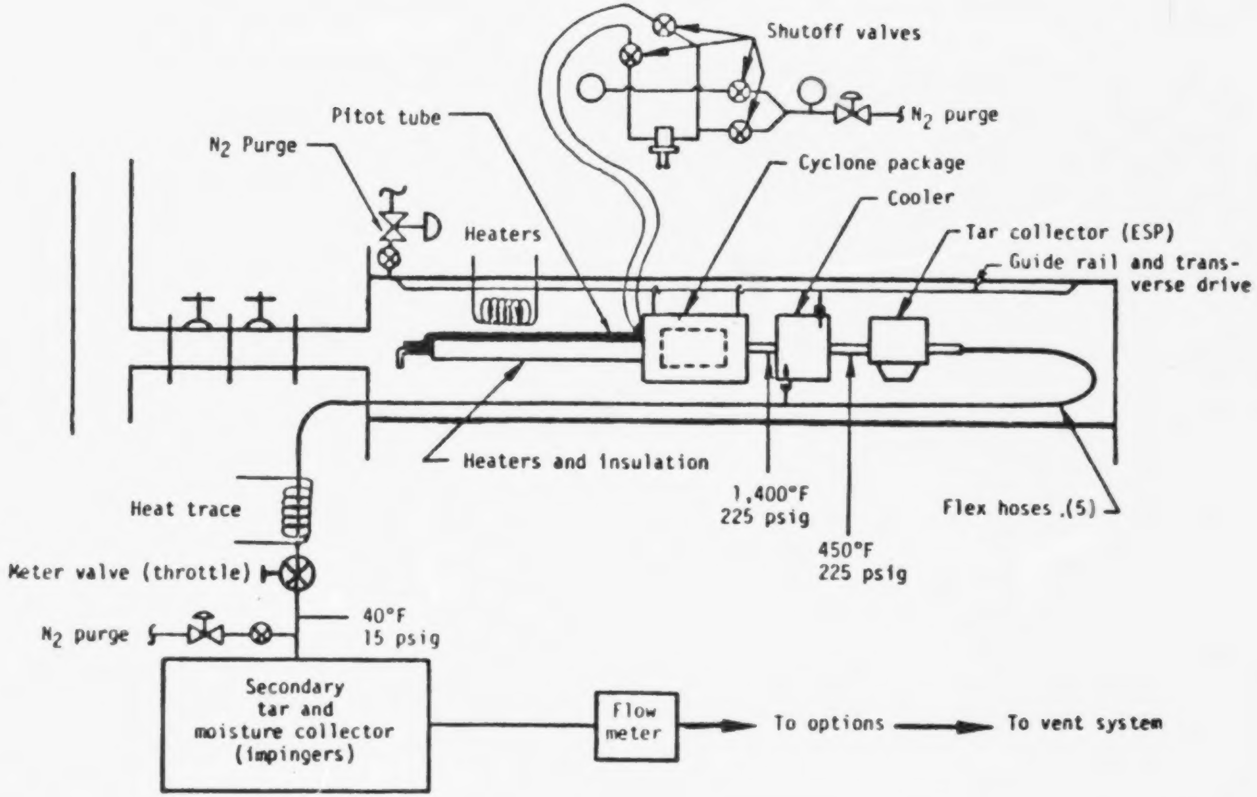
Shutoff valves
Pitot tube
N_2 purge
N_2 Purge
Cyclone package
Cooler
Heaters
Tar collector (ESP)
Guide rail and transverse drive
Heaters and insulation
1,400°F
225 psig
450°F
225 psig
Flex hoses (5)
Heat trace
Meter valve (throttle)
40°F
15 psig
N_2 purge
Secondary tar and moisture collector (impingers)
Flow meter
To options
To vent system

Figure 17. Schematic of gasifier sampling system.

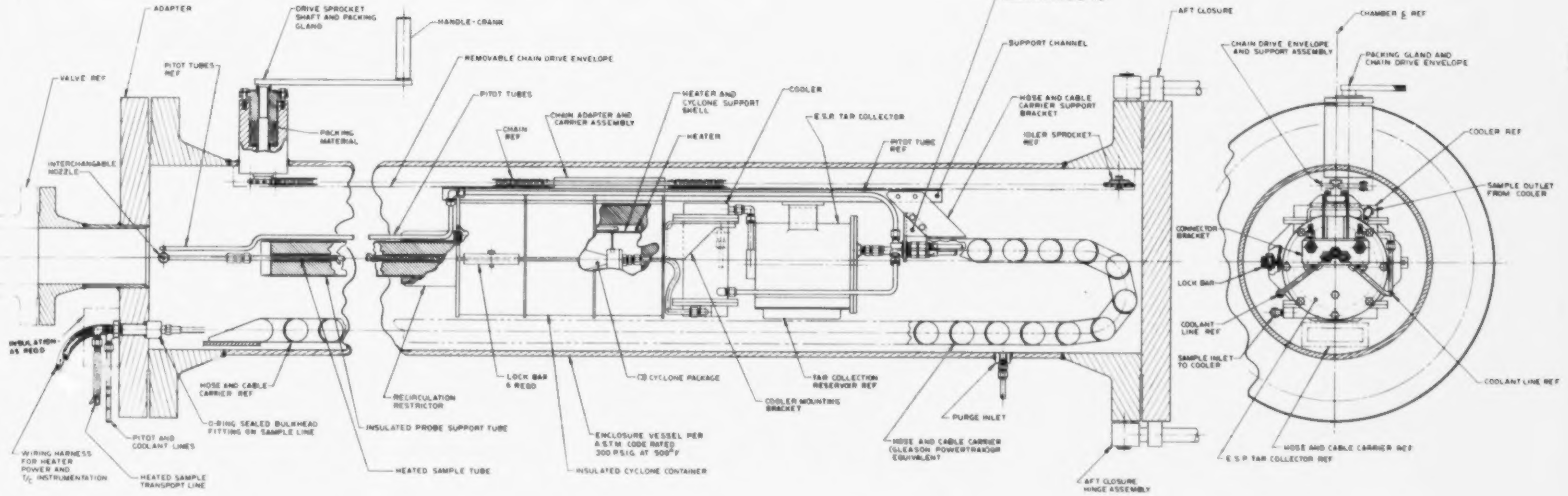
VALVE REF
ADAPTER
PITOT TUBES REF
DRIVE SPROCKET SHAFT AND PACKING GLAND
HANDLE-CRANK
PACKING MATERIAL
INTERCHANGABLE NOZZLE
REMOVABLE CHAIN DRIVE ENVELOPE
PITOT TUBES
CHAIN REF
CHAIN ADAPTER AND CARRIER ASSEMBLY
HEATER AND CYCLONE SUPPORT SHELL
HEATER
COOLER
E.S.P. TAR COLLECTOR
PITOT TUBE REF
CONNECTOR BRACKET REF
SUPPORT CHANNEL
HOSE AND CABLE CARRIER SUPPORT BRACKET
IDLER SPROCKET REF
AFT CLOSURE
INSULATION AS REQD
WIRING HARNESS FOR HEATER POWER AND T/C INSTRUMENTATION
PITOT AND COOLANT LINES
HEATED SAMPLE TRANSPOPT LINE
HOSE AND CABLE CARRIER REF
O-RING SEALED BULKHEAD FITTING ON SAMPLE LINE
INSULATED PROBE SUPPORT TUBE
HEATED SAMPLE TUBE
RECIRCULATION RESTRICTOR
LOCK BAR 6 REQD
ENCLOSURE VESSEL PER A.S.T.M. CODE RATED 300 P.S.I.G. AT 500°F
INSULATED CYCLONE CONTAINER
(3) CYCLONE PACKAGE
COOLER MOUNTING BRACKET
TAR COLLECTION RESERVOIR REF
PURGE INLET
HOSE AND CABLE CARRIER (GLEASON POWERTRAK)OR EQUIVALENT
AFT CLOSURE HINGE ASSEMBLY
CHAMBER ℄ REF
CHAIN DRIVE ENVELOPE AND SUPPORT ASSEMBLY
PACKING GLAND AND CHAIN DRIVE ENVELOPE
COOLER REF
SAMPLE OUTLET FROM COOLER
CONNECTOR BRACKET
LOCK BAR
COOLANT LINE REF
SAMPLE INLET TO COOLER
COOLANT LINE REF
HOSE AND CABLE CARRIER REF
E.S.P. TAR COLLECTOR REF

Figure 18. Conceptual design of gasifier sampling system.

made progressively smaller and the jet velocity becomes progressively greater. In this manner, particulate of smaller size are collected on each stage. A filter is usually placed downstream of the final stage to collect the residual particulate. Table 12 presents a list of commercially available impactors designed for conventional operation. The D_{50}* cut points of each stage are shown.

TABLE 12. SIZE FRACTIONATING POINTS OF SOME COMMERCIAL CASCADE IMPACTORS FOR UNIT DENSITY SPHERES

Stage	Modified Brink	Andersen Mark III	U. of W. (Pilat)	E.R.C. Tag
	0.85 LPM	14 LPM	14 LPM	14 LPM
Scalping cyclone	18.0 μm			
0	11.0			11.1 μm
1	6.29	14.0 μm	39.0 μm	7.7
2	3.74	8.71	15.0	5.5
3	2.59	5.92	6.5	4.0
4	1.41	4.00	3.1	2.8
5	0.93	2.58	1.65	2.0
6	0.56	1.29	0.80	1.3
7		0.80	0.49	0.9
8		0.51		0.6

Cyclones are also inertial collection devices. A cyclone works on the principle of selective sizing through the use of centrifugal forces. In fact, impactors also use the same effect. In a cyclone, a jet of particle laden gas is directed to enter the cylindrical body of the device in a tangential fashion. The momentum of the stream causes the jet to form a spiraling vortex that travels down the inner surface to the funnel-like bottom of the device. The vortex then reverses direction and spirals upward along the centerline with the flow exiting at the top. A sketch of a typical cyclone is shown in Figure 19.

---

*Particle diameter for which the collection efficiency of a given stage is 50 percent

The largest of the particles present are thrown to the outer walls and fall to the bottom of the funnel and are collected in a cup. The fact that large quantities of particulate matter can be collected in the cap is one of the principal advantages of cyclones over impactors. A series of cyclones that are progressively smaller in size can be used to size particles. In general, cyclones are not capable of as small size cuts as impactors and also the size, cut collection efficiency characteristics are generally not as sharp as impactors. A comparison of cyclone and impactor collection efficiency are shown in Figure 20. Again, a final filter is used downstream of the last cyclone stage to collect the residual material.

In some applications, it may not be necessary to classify particulate by size during the test. Filters can be used in cases where only the particulate loading is desired, or when the particulate is of such character that it can be sized optically after collection. Two problems can result in using filters for particulate collection: "blinding" of the filter due to overloading and lack of a suitably efficient filter which can survive at high temperatures. These problems will be discussed further in the following paragraphs.

4.3.2 Suitability for HTHP Application

In some instances, the selection of a particular collection device may not be wholly governed by the collection efficiency or sharpness of the size cut of the device. For example, the pressure housing design of the sampler may dictate the use of a small diameter device such as an impactor or filter instead of a bulkier multiple cyclone system. For example, for the type of sampler design used in the Exxon tests described here, the blowoff loads are proportional to the I.D. of the outer sampler housing. This demands the use of a small diameter device such as an impactor train.

Another important factor governing the selection of a sample collection device is that of the quantity of sample required. For instance, the quantity of particulate that can be collected with an impactor is limited. Once the impactor becomes overloaded with particulate matter, the data can become obscurred by various phenomena. If the stage loading becomes too great, the particulate already collected can become scoured away by the action of particulate continuing to impact the previously collected material. This scouring action can also cause fracture of the already collected material, and the smaller fractured particles can become reentrained back into the system and be collected on lower size cut stages.

Similarly, the use of filters limits the length of time (or amount of sample collected) by virtue of the filter becoming overloaded with particulate. As more particulate is collected, the pressure drop across the filter rises abruptly until the flow is essentially stopped. Once such a condition is reached, there is a great danger of tearing the filter element.

The nonograph presented in Figure 21 is useful in quickly calculating the time required to collect 25 milligrams of material. At heavy dust loading

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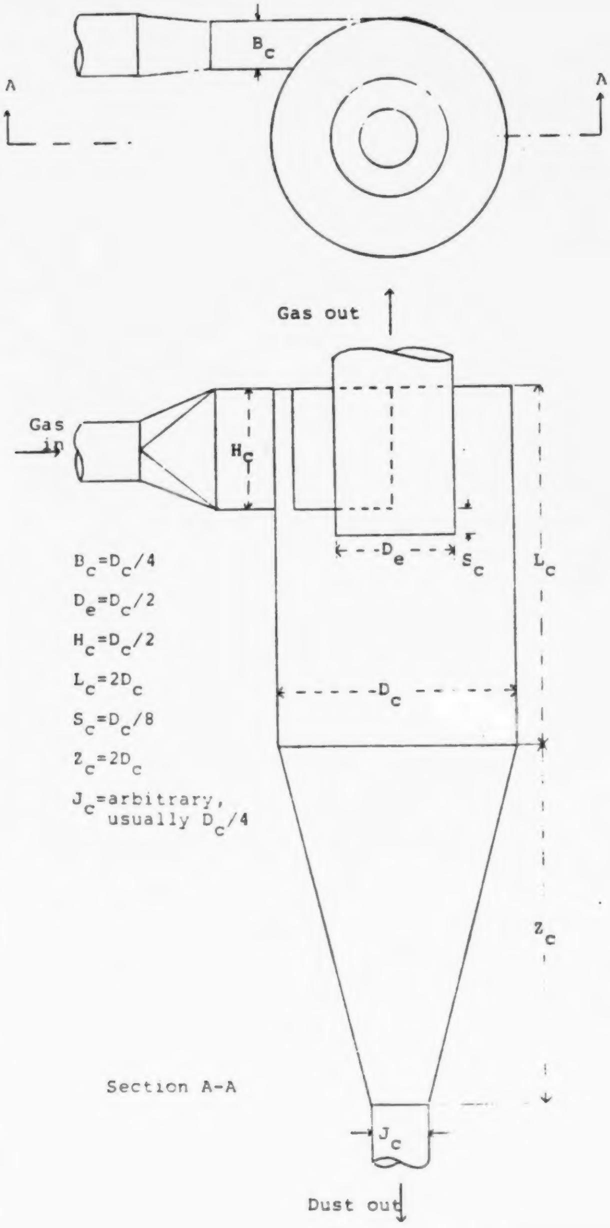
B_c
A
A
Gas out
Gas in
H_c
D_e
S_c
L_c
B_c=D_c/4
D_e=D_c/2
H_c=D_c/2
L_c=2D_c
S_c=D_c/8
Z_c=2D_c
J_c=arbitrary, usually D_c/4
D_c
Z_c
Section A-A
J_c
Dust out

Figure 19. Generalized cyclone design.

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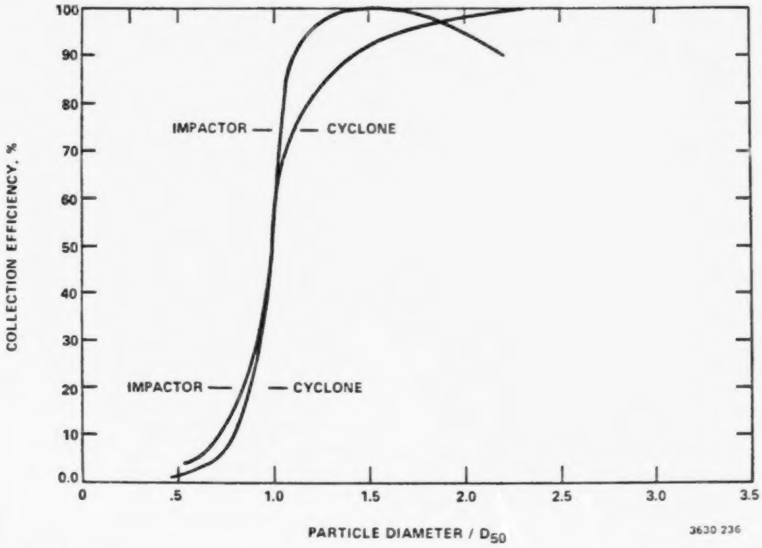


Figure 20. Comparison of cascade impactor stage with cyclone collection efficiency curve (Reference 16).

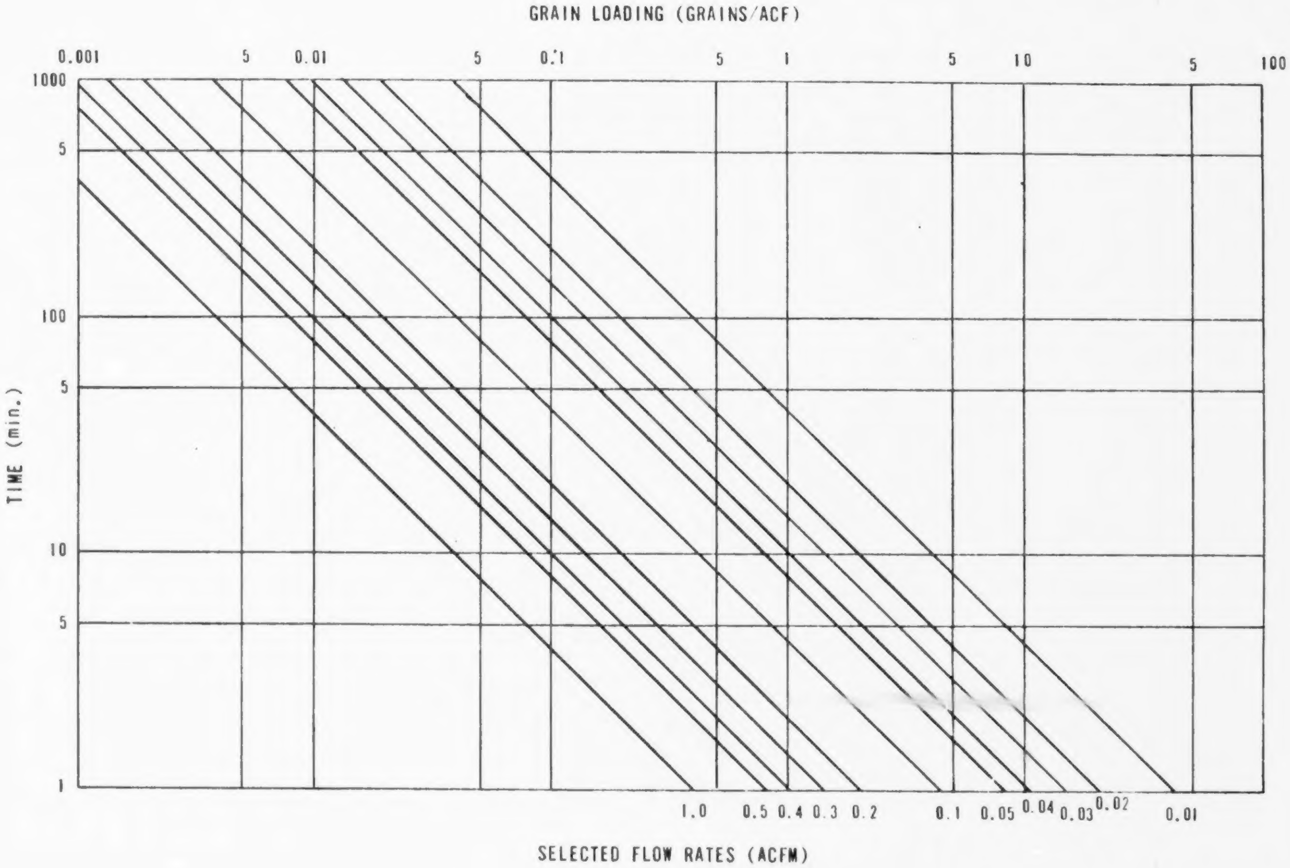


Figure 21. Sampling time determination for total mass collection of 25 milligrams.

conditions, the use of a filter or impactor may be severely impaired by the very short sampling time to reach an overloaded condition. That is to say, to make the sampling time of reasonable length in a high dust loading condition, some means of reducing the loading to the impactors or filters must be found. One approach is to utilize a "scalping" cyclone ahead of the impactor train or filter. The cyclone is designed to take out large size particulate and reduce the particulate loading seen by the collection device. Such an approach is effective in reducing an overloading condition in the upstream stages of an impactor train.

Another criteria for selection is the ability of the device to be insensitive to changes in flowrate through the sampling system. Depending upon the local duct velocity, to maintain isokinetic conditions, the flow through the sampler may be adjusted. Changes in flowrate will affect the performance or any inertial collection device. That is to say, if the device has been designed and calibrated at a given operational flowrate, then excursions from that flowrate will change the size cuts of each impactor or cyclone stage. In general, cyclones are more sensitive to deviations in flowrate, and therefore such changes should be avoided (Reference 16). One means of keeping the flowrate constant while maintaining isokinetic sampling conditions is to change the inlet size. This is most commonly done by using inlet nozzles of various sizes.

Another criteria for selection of a particulate collection device for an HTHP application is its suitability for use based upon environmental considerations. For example, it is common practice to coat each stage of an impactor train with a substrate material (such as grease) to ensure initial adhesion to the collection surface. Most of these materials cannot be used at temperatures much beyond 400°F. Therefore, if one were to attempt to sample at high temperature conditions, the selection of a suitable substrate material may become problematic.

Another example is the selection of a filter material that can survive at high temperatures. It is difficult to find a commercially available filter material that can be used at temperatures above 1000°F. On the other hand, if the stream is cooled prior to particulate collection, certain species may condense. Depending on the stream sampled, the amount of cooling, and the pressure level, condensation of water vapor, alkali metal compounds, and acid gases can result. The presence of these materials in the particulate collection device can obscure the chemical composition of the sample, plug the collection device, or destroy the collector.

A variety of ceramic fiber insulation materials have been tested for filtration applications. Most of those available in mat form are capable of high efficiency collection of fine particles. This is true also of the ceramic paper materials. Properly supported, the fine diameter ($\sim 3\ \mu m$) fibers in these materials will provide absolute filter performance if suitable thickness is used in the filter. Most of the materials contain binders which should be burned out prior to weighing the filter before use to collect a sample. Some also contain shot or beads of ceramic material that remain from the fiber manufacturing process. These beads do not impair filtration performance but may interfere with microscopic interpretation of the dust sample. Of the materials which are commercially available, those produced by ICI and known as SAFFIL fibers tend to contain the least amount of ceramic shot and are therefore a first choice as high temperature absolute filter materials. Sufficient data to select candidate filter materials for test applications is contained in Reference 18.

4.3.3 Operation at High-Temperatures and -Pressures

Cyclones offer the greatest promise for operation at duct conditions in HTHP applications. Experience in calibrating cyclones or impactors at HTHP conditions is lacking, however. Systems must be constructed to calibrate these devices at typical temperature and pressure conditions to fully define their expected performance. There are existing test facilities that have been used for hot filtration testing (at Acurex and Westinghouse) that could be used to perform both high-temperature and high-pressure calibration of sampler collection devices.

Operation at off-calibration conditions can be predicted based upon theoretical considerations of the variations in collection mechanisms with temperature and pressure. Properties of gases at high temperatures and pressures can be found in Reference 9.

4.4 SAMPLE HANDLING AND FLOW MEASUREMENT

In this section source general considerations regarding the choice of design features upon sample collection are discussed. These include the effects of isokinetic sampling, flowfield measurement, flow conditions within the probe, and material compatibility.

4.4.1 Isokinetic Sampling Considerations

To accurately sample in a particulate laden stream, a representative mass of particles must be withdrawn from the gas at the same velocity as the particles are flowing in the gas stream. This condition, where the velocity at the probe inlet face is equal to the local stream velocity, is known as isokinetic sampling. The theoretical aspects of isokinetic sampling are discussed in References 10 and 11.

To sample under isokinetic conditions, two approaches have been used. The first is to use inlet nozzles of various sizes for different stream velocities while the flowrate through the sampler remains constant. In addition, a continously variable two-dimensional inlet nozzle could be used to avoid changing the nozzle size every time a new location in the duct is to be sampled. This approach has not be in common use. Cyclone performance is particularly sensitive to changes in flow and individual cyclone cut points vary with velocity, therefore, the rate of sample extraction should be nearly constant.

The second approach to maintaining isokinetic conditions is to use a fixed inlet nozzle and vary the flowrate through the sampler. Within a narrow range this can be done even for cyclone operation, but if the flowrate variation is considerable cyclone operation will be affected. This can be overcome by using a bypass flow system so that the cyclone always receives a constant volume flow. This becomes complex and difficult in that each leg of the bifurcated stream must be measured individually and particulate biasing could occur. For high-pressure applications no pump is required, instead an orifice/valve combination can be used to throttle the flow to atmospheric conditions.

4.4.2 Automatic Isokinetic Sampling

To determine the stream velocity for isokinetic sampling by ordinary means it is necessary to determine the stream dynamic pressure differential, stream static pressure and temperature. The latter two quantities, along with gas composition information, are necessary to determine the gas density so that the velocity can be obtained through the Bernoulli equation.

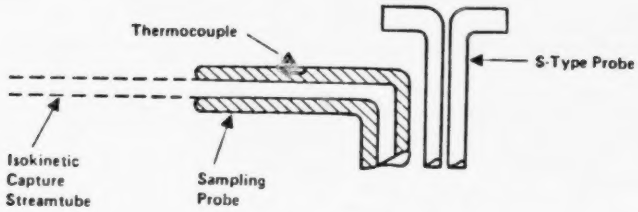
Potentially, there are automatic approaches to sampling isokinetically. Figure 22 shows three of these approaches. In Figure 22(a), the approach is conventional in that the stream differential pressure, pressure, and temperature are obtained. These are input to an analog logic circuit or minicomputer that determines the required sampler flowrate and the flow controller is set accordingly. The approaches shown in Figure 22(b) and 22(c) do not require that the stream density be determined. The principal of operation requires that the static pressure of the stream be determined by a field probe. At the same time the static pressure at the inlet port is also measured. When the static pressures at each location are equal, the velocities are also equal and isokinetic conditions will exist.

In developing an isokinetic flow control system, whether manual or automatic, it is necessary to select differential pressure transducers that are sufficiently precise to maintain isokinetic conditions to within 10 percent. One can compute the required transducer sensitivity by calculating the allowed error signal which will produce a predetermined error in the velocity.

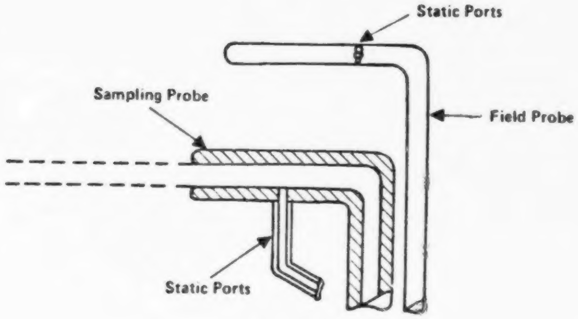
4.4.3 Other Considerations

When making measurements near bends in ducts or downstream of sudden expansions or contractions, care should be taken in locating the sample extraction site. Ideally, the sampling location should be 8 to 10 duct diameters downstream and two diameters upstream of such flow disturbances to ensure that the stream is nearly uniform and well mixed. As piping costs are high, long straight runs ideal for particulate sampling may not be available. When this is the case, flow baffles followed by flow straighteners may be used to promote uniform distribution of the velocity and particulate fields.

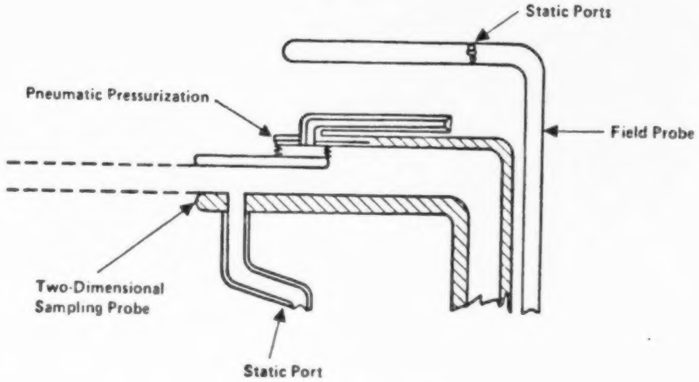
In designing the internal flow system of the sampler, care should be taken to examine the distribution of temperature and pressure. That is, as the gas flows through the probe it should not be subjected to any sudden changes of temperature or pressure. For example, if locally within the probe the flow undergoes an abrupt pressure drop, local supersonic flow conditions might occur. Should this occur the flow will expand locally to supersonic Mach numbers and a sharp drop in local stream static temperature will occur.


Thermocouple
S-Type Probe
Isokinetic Capture Streamtube
Sampling Probe

a) Analytical feedback


Static Ports
Sampling Probe
Field Probe
Static Ports

b) Null balance


Static Ports
Pneumatic Pressurization
Field Probe
Two-Dimensional Sampling Probe
Static Port

c) Variable inlet

Figure 22. Automatic isokinetic sampling probes.

This may cause a phase change of some of the chemical species which may be present. Such design precautions require careful calculation and examination of the flow system in the design phase.

Finally, in selecting materials for the probe care should be taken that they are compatible with the gaseous species present. For example, special care should be taken in selecting nonmetallic materials for seals and valve seats. Many of these nonmetallic materials are subject to attack by various gases present or acids formed through the combination of acid gases and condensed water vapor. Reference 17 discusses materials for use in HTHP environments.

4.5 GENERAL FINDINGS AND EXPERIENCES DERIVED FROM EXXON TESTS

The following discussion presents a review of the test results obtained from the two test series conducted at the Exxon Miniplant. A more detailed discussion of the entire test program is contained in Reference 6. After reviewing the general results of the sampling tests performed at the Exxon PFBC at Linden, New Jersey, a discussion follows that points to some experiences that were encountered prior to and at the time of the tests. This discussion is presented in an effort to show the types of problems that may be encountered in future HTHP testing so that others may learn from our experiences.

4.5.1 Test Results -- Phase I and II

In Section 2.1 of this report the activities and hardware development leading to the actual testing was discussed. A detailed discussion of the equipment, including drawings, is contained in Reference 6.

To reiterate, two different test series were conducted: Phase I and Phase II. During the Phase I tests the objectives were to demonstrate that the sampling approach taken was a viable one from the standpoint of data accuracy and repeatability and that the sampling system was safe and reliable in use. During test Phase I three sampling runs were made: one (Run 2) using a thimble filter to collect the total particulate catch and two additional tests (Runs 2 and 3) using a cascade impactor (containing seven stages) followed by a final filter. The following types of data were recorded during the Phase I tests:

- Particulate size distribution
- Particulate chemical composition
- Particulate shape
- Particulate concentration
- Process temperature and pressure
- Moisture content
- Structural temperatures (valves and probe housing)
- Trace element samples
- Organic samples

Phase I Tests

Table 13 shows the test conditions present during the three runs comprising Phase I testing. The bed temperature was typically 300°F higher than the duct temperatures at the sampling location. Typical wall temperatures recorded during Run 1 are shown in Table 14. The total particulate concentration from the other test runs are shown in Table 15. Here the most accurate reading (1.58 g/m^3) comes from Run 1 where the bulk filter was used. This is because a longer sampling period was used and bulk filter catches are less subject to cumulative errors associated from weighing the catches from the eight stages of particulate catch in test Runs 2 and 3. A weight breakdown by impactor stage plus final filter particulate collected during Runs 2 and 3 is presented in Table 16. A cursory examination of this table shows that the predominance of particulate collected was in the 1-10 μm range.

Figure 23 summarizes the results of Runs 2 and 3 of Phase I testing. Particulate distributions ranged from 0.3 to 30 μm in aerodynamic diameter. These curves were constructed directly from the D_{50} cut points of the series impactors used. A temperature correction for the operating temperature of 450°F was applied based on theoretical considerations. The effect of pressure on the operation of the series impactors was determined to be small based on theoretical considerations. The repeatability of the data as exhibited by Figure 23 was deemed to be good for a first demonstration. Figure 24 shows the particulate collected on the substrate material during Run 3. A closer examination of stage 3 in Figure 25 shows the classic "pyramids" of particulate collected.

The particulate sample from Run 2 was subsequently examined using a scanning electron miscrscope. An example of the particulate is shown in Figure 26. From this photo it is evident that the particulate is somewhat fused together so it is difficult to correlate the physical size to the impactor cut size. The samples were chemically analyzed using a dispersive X-ray fluorescence analyzer. Later a SSMS examination was performed in the analysis of Phase II results and Phase I, Run 1, results as will be explained below.

Phase II Tests

A total of four test runs were made during Phase II of testing. The samples from Run 3 of Phase II were retained for SSMS analysis. Table 17 shows the results of the SSMS analysis from Run 3 of Phase II. Examination of the table shows that the quantities of material obtained on the downstream filter were inadequate to make a meaningful comparison to the quantities contained in the scalping cyclone and upstream filter. This was because the upstream filter removed a great bulk of particulates. In addition the downstream filter was subject to some contamination as will be discussed later. To resolve the problem of lack of sufficient sample quantity a comparison was made with the chemical composition of the bulk filter catch from Run 1, Phase I. Since the test conditions were virtually identical the results should be, it was reasoned, equally valid.

TABLE 13. PHASE I TEST CONDITIONS

Run:	#1	#2	#3
Date	3-31-77	4-1-77	4-1-77
Time	3:30 p.m.	10:30 a.m.	2:40 p.m.
Ambient temperature	18°C (64°F)	21°C (70°F)	19°C (67°F)
Bed Conditions			
Temperature	900°C (1650°F)	900°C (1650°F)	900°C (1650°F)
Pressure (gage)	912 kPa (9 atm)	912 kPa (9 atm)	912 kPa (9 atm)
Ca/Sulphur Ratio	1.25	1.25	1.25
Excess Air	30%	30%	30%
Coal	Champion	Champion	Champion
Dolomite	Pfzizer	Pfzizer	Pfzizer
Flowrate - sm^3/min (scfm)	14(544)	14(546)	14(546)
Average Duct Velocity - m/s(ft/sec)	2.0(6.7)	1.9(6.3)	2.0(6.7)

TABLE 14. STRUCTURE TEMPERATURES

Time	Valve-Probe Side	Inner Probe Housing	Outer Probe Housing
0	64°C(147°F)	56°C(133°F)	56°C(133°F)
10 min	71°C(159°F)	55°C(131°F)	68°C(154°F)
20 min	69°C(157°F)	56°C(133°F)	75°C(167°F)
60 min	88°C(190°F)	N/A	N/A

TABLE 15. PARTICULATE CONTENT

	Run #1	Run #2	Run #3
Date:	3-31-77	4-1-77	4-1-77
Time:	1530	1030	1500
Particle Catch: (grams)			
Filter	3.2515	-	-
Impactor	-	0.0554	0.0892
Residue	1.8565	0.0334	0.0595
Total	5.108	0.0884	0.1497
Sample Volume: m^3(scf)	3.22(122.5)	0.082(3.13)	0.132(5.03)
Particle Content: (g/m^3)(gr/scf)	1.58(0.64)	1.06(0.43)	1.13(0.46)
Particle content: (g/m^3)(gr/scf) (Anisokinetic Correction Applied)	1.60(0.65)	1.08(0.44)	1.16(0.47)

TABLE 16. PARTICLE SIZE DISTRIBUTION

		Run #2			Run #3		
Stage	D_{50} Microns	Weight Collected	% Total Weight	% Smaller	Weight Collected	% Total Weight	% Smaller
1	26.0	0.0076	13.7	86.3	0.0093	10.4	89.6
2	12.0	0.0080	14.4	71.8	0.008	9.0	80.6
3	4.3	0.0171	30.9	41.0	0.0221	24.8	55.0
4	2.1	0.0139	25.1	15.9	0.0215	24.1	31.7
5	1.2	0.0022	4.0	12.0	0.0135	15.1	16.6
6	0.6	0.0036	6.5	5.4	0.0081	9.1	7.5
7	0.3	0.0020	3.6	1.8	0.0039	4.4	3.1
Filter		0.0010	1.8		0.0028	3.1	
		0.0554 grams			0.0892 grams		

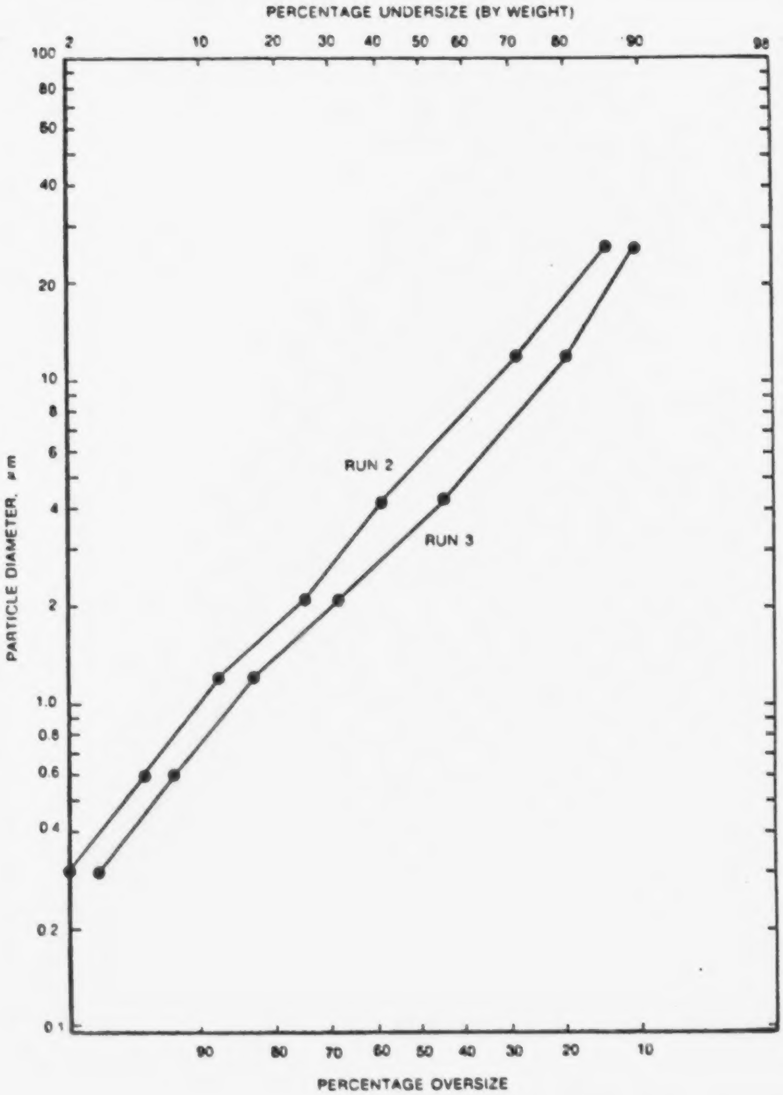


Figure 23. Particle size distribution -- Phase I tests.

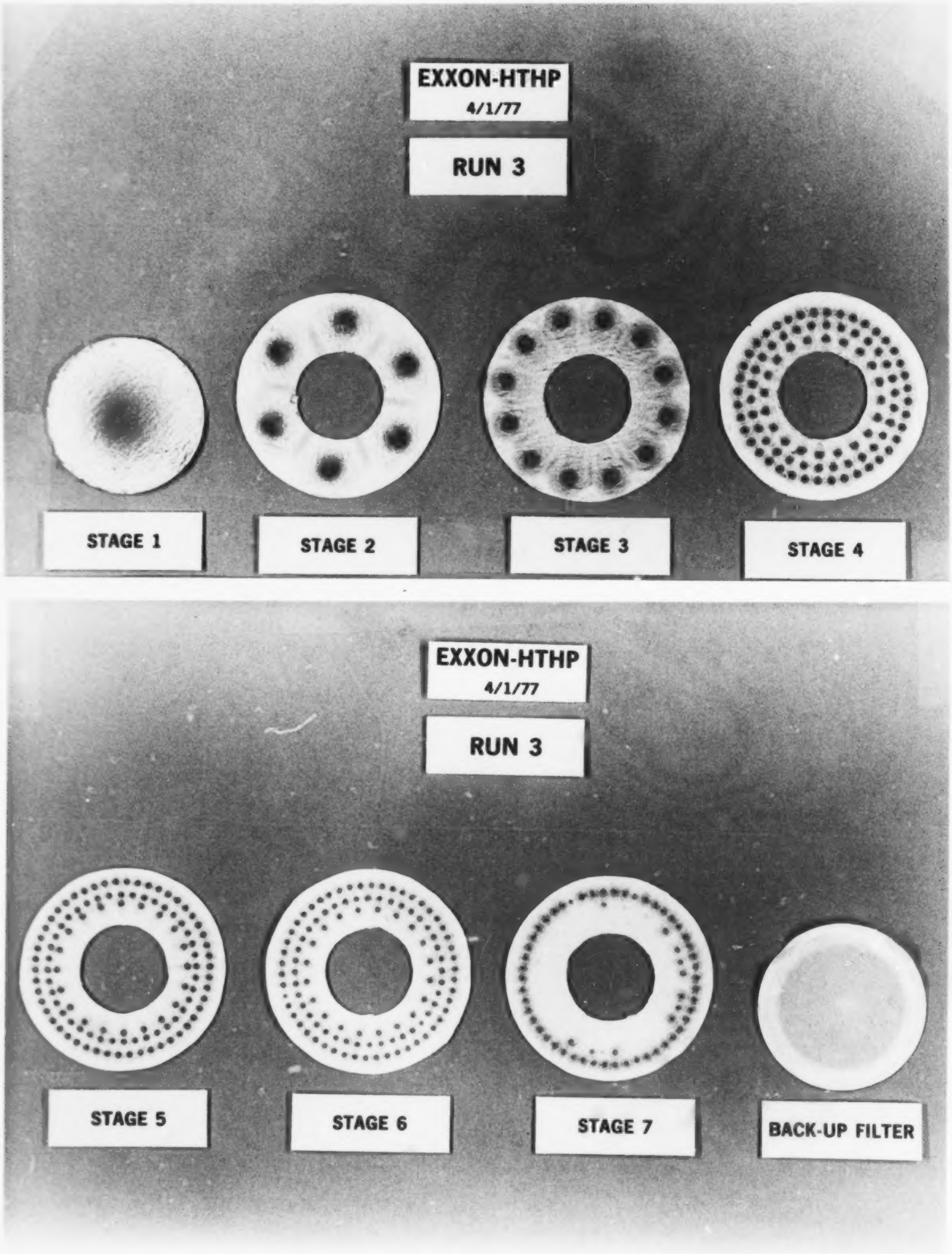

EXXON-HTHP
4/1/77
RUN 3
STAGE 1
STAGE 2
STAGE 3
STAGE 4
EXXON-HTHP
4/1/77
RUN 3
STAGE 5
STAGE 6
STAGE 7
BACK-UP FILTER

Figure 24. Impactor substrates.

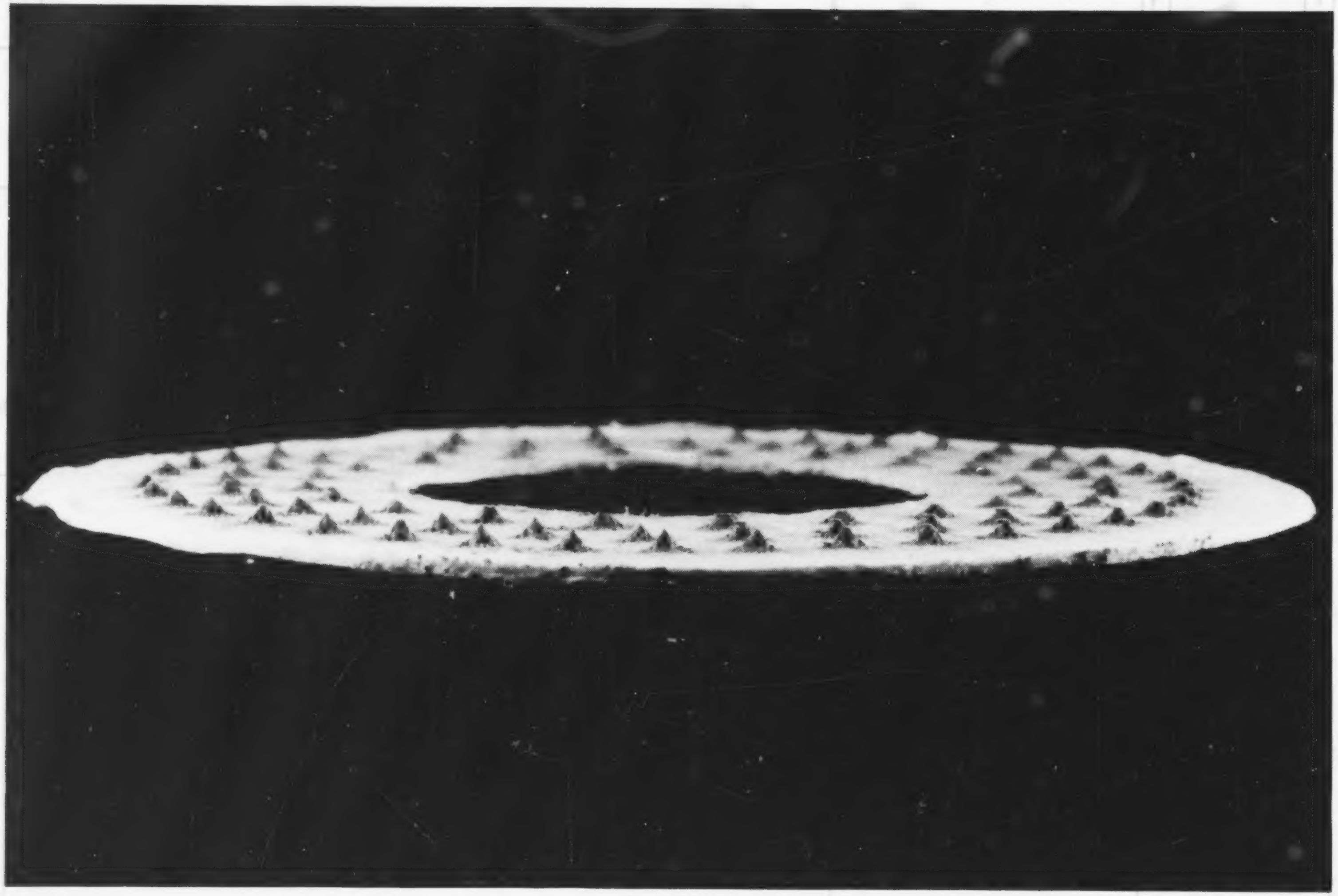
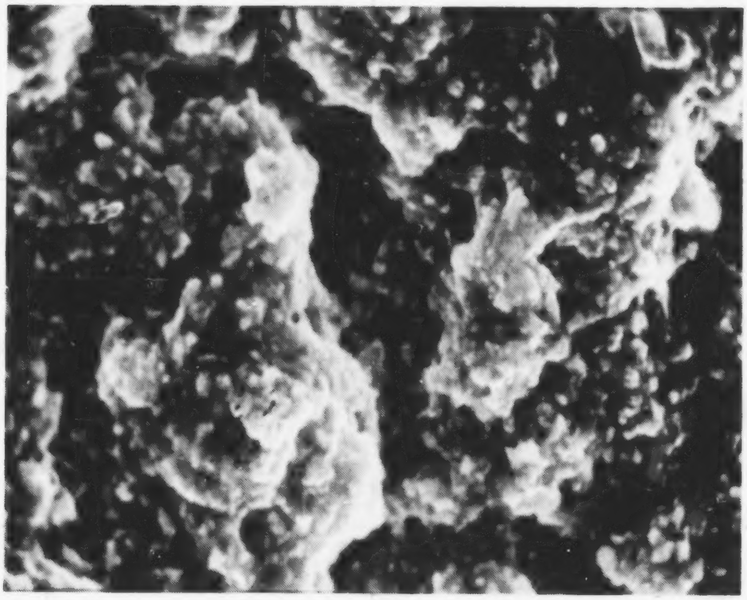
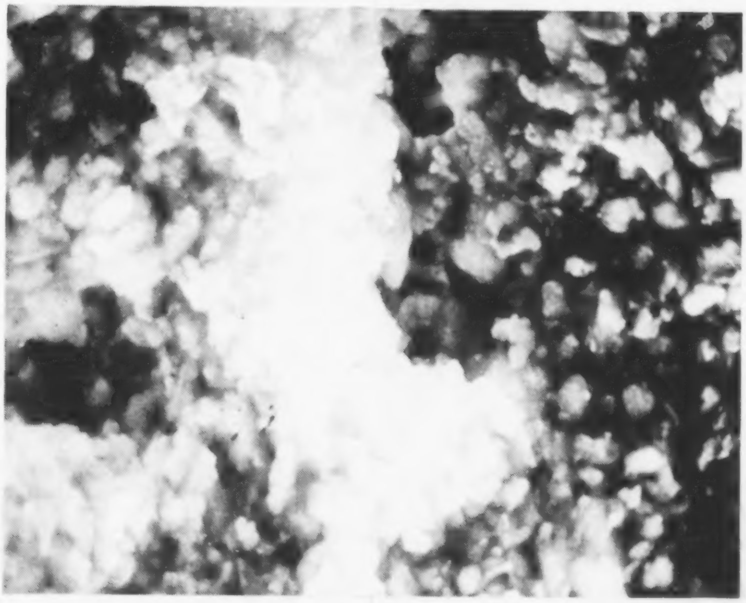


Figure 25. Impactor substrate Run 3, Stage 5.



1000X

|←→| 10 microns



3000X

|←→| 3 microns

Figure 26. Particle photomicrographs, Stage 4.

TABLE 17. CONCENTRATION OF ELEMENTS IN FLYASH SSMS ANALYSIS (PARTIAL)

Element	TEST #3 - PHASE II				PHASE I
	Cyclone (ppm)	Front (ppm)	Rear (ppm)	Rear Blank (ppm)	Bulk Filter (ppm)
K	8,200	8,850	15.1	5.4	16,250
Na	1,310	2,500	< 135. *	91.	3,560
Rb	< 70	< 68	< .43 *	.13	866
Cs	6.7	.23	< .62 *	------	8.8
Al	164,000	94,000	64.2	6.4	Major
Si	94,000	82,600	<2510. *	1210.	310,000
Fe	30,000	13,400	60.	2.36	36,300
Ca	20,000	19,000	< 6.6 *	7.3	44,000
Mg	11,400	17,800	38.	5.9	28,600
Ti	2,430	1,950	7.1	3.1	10,600
Sr	810	555	.6	1.15	1,320
Ba	710	694	< 5.0	1.5	1,080
Au	650	118	< 1.4	------	213
P	276	223	< 224. *	68.	1,880
Cu	248	165	< 1.5 *	.91	142
Zr	160	140	< 2.1 *	.64	334
Ni	120	100	5.8	.29	348
Cr	< 90	< 140	< 13.1	6.4	366
Pb	85	75	< 4.0 *	1.2	86

Notes: < - natural background limited detection limit

<* - blank limited detection limit

To reiterate the major objective of the Phase II tests was to determine if alkali metal compounds would condense out of the stream if the sample was cooled to 450°F. Table 18 shows a summary of the results obtained from Phase II tests. The results are shown in a dimensionless form, normalized by the iron and magnesium content of the coal.

A comparison is made of the ratio of nondimensional quantities caught "hot" and caught "cold." These results showed no consistent evidence of alkali metal condensation occurring as a result of sample cool down. The results are not totally conclusive, however, and the amount of data is limited. To avoid future uncertainties arising over whether the sample can be cooled prior to collection, it is advised that in most future test programs the particulate be collected at duct conditions. This will require the development of particulate collection devices that can be operated and calibrated at high-temperature and -pressure.

In summary, the Exxon tests produced good results and demonstrated that the sampler design worked well. In the course of obtaining these positive results several considerations and experiences were identified that require discussion.

4.5.2 Experiences Derived from Exxon Testing

In the course of planning and performing the tests at the Exxon Miniplant the following problem areas were identified.

Probe alignment problems were encountered during the first test phase. Such problems can be expected when the probe is very long and the clearances through the isolation valves are tight. The S-type pitot probe tube was damaged during insertion. Hydraulic actuation makes the danger of such damage greater since all "feel" is gone.

Double gate valves were used for probe isolation during the Exxon tests. It is felt that a double valve approach is safer and increases system reliability. During Phase I testing, some problems were encountered from the leakage past the valves. The problem was narrowed down to the fact that particulate was accumulating in the bottom of the value seat with the gate moving down vertically into the seat. The problem was solved by rotating the valve so that the particulate did not accumulate in the bottom of the valve seat.

During the condensation tests (Phase II), a flow control valve was located upstream of the downstream (cold) filter which was outside of the probe housing. A problem was encountered when the heat tracing on the tube downstream of the valve was unable to compensate for temperature reduction due to the throttling effects of the valve. As a result, the catch in the downstream filter was contaminated with sulfuric acid condensate because of the sudden drop in temperature. A subsequent analysis of sulfuric acid condensation limits bore out this conclusion. In addition, the throttling valve seat material was apparently attacked by the sulfuric acid that further contaminated the downstream filter. As a result in future tests care should be taken to ensure that abrupt temperature drops do not occur and that all

TABLE 18. PARTIAL COMPARISON OF FRONT AND REAR PARTICULATE CATCHES FROM EXXON TEST SERIES I AND II

	Phase I Tests	Phase II Tests			
	Bulk Filter	Cyclone	Front Filter	Avg.	Ratio
$\frac{K}{Fe}$	0.45	0.27	0.66	0.47	0.96
$\frac{Na}{Fe}$	0.10	0.04	0.19	0.12	0.83
$\frac{Si}{Fe}$	8.54	3.13	6.16	4.65	1.84
$\frac{Ca}{Fe}$	1.23	0.67	1.42	1.05	1.17
$\frac{Mg}{Fe}$	0.79	0.38	1.33	0.86	0.92
$\frac{Sr}{Fe}$	0.04	0.03	0.04	0.04	1.00
$\frac{Ba}{Fe}$	0.03	0.02	0.05	0.04	0.75
$\frac{K}{Mg}$	0.57	0.72	0.50	0.61	0.93
$\frac{Na}{Mg}$	0.12	0.11	0.14	0.13	0.92
$\frac{Si}{Mg}$	10.84	8.25	4.64	6.45	1.68
$\frac{Fe}{Mg}$	1.27	2.63	0.75	1.69	0.75
$\frac{Ca}{Mg}$	1.56	1.75	1.07	1.41	1.11
$\frac{Sr}{Mg}$	0.05	0.07	0.03	0.05	1.00
$\frac{Ba}{Mg}$	0.04	0.06	0.04	0.05	0.80

valve seat materials be carefully selected for compatibility with the species present.

Finally, the question of safety cannot be overemphasized in HTHP sampling. Fortunately, no severe safety problems occurred during the Exxon tests. It should be recalled that if large system leaks were to occur the gases escaping would accelerate to supersonic velocities upon expansion. Not only are the hot escaping gases dangerous but the erosive effects of the particulate exiting at these high velocities could be disastrous to persons or equipment in the area.

In future tests at coal gasification facilities, the safety problems are even greater. In addition to the problems already mentioned, if a leak occurred the danger of an explosion is very real. Also, many of the constituents present may be carcinogenic depending upon the sampling location in the process.

4.6 BEYOND PROTOTYPE SYSTEMS

The sampling system designed and tested in this program exemplified the prototype approach. The design of the probe hardware utilized approaches employed to ensure proper operation in a first-of-a-kind sampling exercise. Now that the general approach has been demonstrated at least at an PFBC facility we can look to some of the questions affecting the development of more general purpose sampling equipment designed to meet a wider variety of applications. It should be emphasized that to some extent this discussion is premature in that prototype sampling systems have not yet been demonstrated in coal gasification or MHD applications.

4.6.1 Generalized Sampling Systems

One might conjecture that it would be valid to assume that a general purpose HTHP sampling system could be constructed that would serve a wide variety of purposes. Indeed such systems as the SASS are used in a variety of ways in conventional sampling applications. This approach is not entirely viable for HTHP coal conversion system applications.

For example, temperatures and pressures may vary from 1000 to 3000°F and 1 to 100 atm depending upon the application. The wide range of temperatures (and for that matter pressures) will dictate when the probe surfaces must be protected by cooling, since the temperature, pressure and velocity strongly affect the heat transfer coefficient. Therefore, over a wide range of conditions the cooling requirements may be drastically different. Also the pressure level (and for that matter temperature) will dictate the pressure vessel design considerations. Consequently, from the standpoint of pressure and temperature, the system would have to be considerably overdesigned with respect to cooling (if required at all, in many applications) and pressure vessel strength.

Gaseous constituents would greatly dictate the choice of materials. Gases present in reducing environments are considerably more corrosive to metal parts than those found in oxidizing environments. These also combine with operating temperature and pressure levels in selecting probe materials. Consequently, material for a generalized probe would be of an exotic nature to cover the most extreme conditions.

Other considerations would tend to thwart the design of a general purpose sampling system. If a general purpose sampler is designed it must be capable of traversing over a wide variety of duct sizes ranging from a few inches to several feet. This is a particularly awkward situation that confronts a generalized system approach. If a probe is designed to traverse a maximum 5-foot duct it is likely to be oversized to sample in a one foot duct. Constraints such as the total available external operating envelope are likely to restrict the use of an oversized probe system in many applications.

Duct velocity and particulate loading are other parameters which make the generalized design approach difficult. If the probe and particulate collection devices are designed for some maximum anticipated condition then when sampling conditions at the opposite end of the spectrum, nozzle sizes, isokinetic considerations, and sampling times are likely to become untenable. For example, if the particulate collection device is designed to collect particulate at a high dust loading and high velocity condition in a matter of a few minutes of sampling time, then it might take hours to collect a representative sample at a low dust loading, low velocity condition.

Based upon the arguments delineated above it is easy to see why each HTHP sampling application demands a custom approach to some extent. It is therefore unlikely that a single sampling system can be used in all or even most PFBC applications for example. This is not to say that a given probe design can only be used in a single application, facility, or location in the process stream. It is also likely that a given design can have multiple uses. But it is impractical to consider the generalized design approach for HTHP applications. This explains one of the inherent reasons that costs for producing a HTHP sampling system are high.

4.6.2 Modular Approach

There is another approach to the design and fabrication of HTHP sampling systems that offers a means of reducing costs. Rather than consider a generalized design approach, a modular approach can be taken. In the modular approach the design aspects of each of the major sampling system components (probe housing, traversing mechanism, collection devices, cooling system, flow control system, etc.) can be refined to a point where sampler design can be simplified.

Design guides for each of the major systems would be developed, calibrated, and tested separately. Principals would be defined for extrapolating these designs to other situations. For example, a comprehensive program to develop and calibrate HTHP cyclones over a wide range of operating conditions would allow the designer to quickly select a design applicable to his particular set of conditions. Similarly, for coal gasification applications a program could be developed to perfect a tar collection device with design parameters identified so that the probe designer could match his application. In this manner, each new application would become less of a new development program and more of a predictable design exercise. A modular approach could not be full proof to the point where acceptance tests would not be required, however.

To some extent the modular approach was demonstrated during the Exxon tests. The internal and external configuration of the probe was modified from the original using the impacter train to the Phase II configuration that employed the scalping cyclone and dual filter arrangement. This was a good example of the versatility of the modular approach.

4.6.3 Other Desirable Features

In the course of the period of performance of this contract at least two process developers expressed the desire to have one or both of two automatic features. These were (1) an automatic isokinetic capability and (2) an automatic traversing capability. In both cases the reasons for these desired features were to (1) improve system accuracy (2) lessen sampling time and (3) reduce the number of required operators.

For long term process development situations at least one process developer felt that the trade of the cost of automatic systems would be cost effective in terms of reducing the operator labor required. This will not always be the case. But once the automatic systems are developed their use may become cost competitive in a wider variety of applications.

SECTION 5

REVIEW OF PARTICULAR PROBLEMS ASSOCIATED WITH HTHP SAMPLING

In the previous sections several problem areas pertaining to HTHP sampling were discussed. In this section a few of those areas will be reviewed and elaborated upon. Based upon the current state of HTHP sampling, some recommended solutions to these problems are offered.

5.1 PFBC SAMPLING

Two areas defined as potential problem areas based upon direct experience gained in this program are (1) pressure containment and (2) condensation effects.

A satisfactory means of containing high pressure levels ensuring safety and ease of operation remains a problem. Based upon the experience gained in actual tests and studies performed during the period of this contract, a proposed means of pressure containment has evolved. In future sampler designs where traversing is required, it is proposed that the best means of containing high pressures is by use of the total enclosure concept as discussed in Section 4.2. The elimination of all sliding seals and power-driven traversing mechanisms make this approach very attractive. The technique remains to be proven in practice, however. There are potential problems relating to maintenance of the internal components, and these must be put to test in practice.

Some problems of probe condensation effects were encountered during the Exxon tests. At times, controlled condensation might be desirable to remove water vapor or acid gases. The question of alkali metal condensation remains open to discussion. Special care should be taken in analyzing and designing the internal flow system so that pressure and temperature distributions can be determined beforehand. If abrupt temperature drops are unavoidable, then flow passages should be adequately heat traced to avoid condensation. The best approach is to collect the particulate at stream conditions then drop the pressure and temperature after collection.

5.2 GASIFIER SAMPLING

The problem areas discussed in Section 5.1 pertain equally to gasifier sampling. In fact, these problems are accentuated in some gasification processes. As discussed earlier, the operational pressure levels in some high Btu gasification processes far exceed those in PFBC processes. Therefore, pressure containment is a very difficult problem for sampling in gasification facilities.

In addition, as discussed previously, condensation of volatiles from the gasification process presents a problem not only from the standpoint of collecting samples, but also as it relates to the pressure containment problem. For example, sliding seal approaches (see Section 4.2) may prove untenable in gasifier environments. The seals would be subject to attack from the acid gases present, and tar condensation could foul sliding seal surfaces. Hence, it appears that the best approach to pressure containment for gasifier sampling is the total enclosure approach. Again, this recommendation is subject to verification by test.

Of course the other major problem confronting the design of a gasifier sampling system is that of collecting tar mist separate from the solid particular matter. Some of these problems were discussed in Section 2.2. Reference 4 discusses the various approaches considered to collect the tar mist once it has been condensed from the stream. Again, the approach would be to maintain the sample at the process stream temperature and pressure where the volatiles are in a vapor state, collect the solid particles, then drop the temperature and pressure to form a tar mist and subsequently collect the tar mist. Most of the devices (scrubbers, impingers, cyclones, filters, etc.) for collecting the tar mist mentioned in Section 2.2 and discussed in detail in Reference 4 are not efficient and are subject to plugging. The exception would be the ESP that has been successfully used to collect tars in large industrial applications. The condensation properties of tars must be better defined to properly design a small ESP collector. Work sponsored by DOE has been underway for some time at the University of California at Berkeley to characterize the chemical and condensation properties of gasification tars (DOE Contract DE-AC01-79ET14-884, Dr. J. M. Prausnitz -- UCB).

5.3 OTHER APPLICATIONS

Two other possible applications for HTHP sampling were mentioned in this report: MHD exhaust sampling and catalytic cracker sampling. Both of these areas present new challenges for HTHP sampling technology.

For MHD sampling the major problem is sampling at high temperature (2200°F) in an erosive environment. Also the application calls for a fixed probe approach. The recommended solution was to make the sample tube and cyclone collection device of ceramic material. The ceramic would be temperature and erosion resistant. The ceramic fabrication approach offers promise for other applications also. However, ceramics are subject to thermal shock failure and means must be devised to avoid such a consequence. Again, prototype development of a ceramic system should go far to prove the concept.

The other application, sampling at a catalytic cracker, is a lower temperature application. Here ceramic fabrication techniques are not required. Although fixed point sampling is the only requirement the high velocity erosive environment dictates that the probe be removed from the stream while not in operation. Here the proposed solution as discussed in Section 4.2 is to use the semifixed housing design approach.

SECTION 6

CONCLUSIONS AND RECOMMENDATIONS

The scope of the program undertaken was very comprehensive. The major effort that of the demonstration of HTHP sampling at the Exxon Miniplant was successful. The design and testing of this apparatus provided valuable insight into some of the problems of HTHP sampling.

The design, analysis, and testing of an HTHP sampling system can be an expensive proposition. The system components are complex and constant attention must be placed upon compatibility the process stream constituents and safety aspects. Short cut approaches to HTHP sampling are not advised.

In addition to the conclusions drawn above, the other portions of the program especially the SOA review led to several conclusions. At the time of the survey of process developers not many had begun to consider their needs for HTHP particulate sampling. As their systems approach further states of development especially with respect to cleanup devices, a clearer list of sampling requirements will develop.

Some special problems were identified during the SOA review. One problem that must receive further attention is that of development of system components for coal gasification sampling. The selection of materials compatible with gaseous constituents poses only one problem. Tar separation and collection presents another problem area which must be addressed prior to taking the next big step to coal gasification sampling. Perhaps a conservative approach would be to develop a sampling system designed for operation at an atmospheric gasifier. After a successful demonstration further development for sampling at both high temperature and pressure could be considered. Safety considerations for gasifier sampling should receive foremost attention to ensure the safety of the operators and the plant equipment. Perhaps a separate program aimed at the issue of safety for this application is appropriate.

Another area that requires further attention is that of modularization of design. For most applications the HTHP sampler design tends to be customized to the particular application. However, certain components such as pressure vessels, impactors, cyclones filters, ESPs, etc., could be designed and laboratory tested as individual components. Once these basic components are developed, deviations for particular applications would become more routine. This modular approach in which separate components would be designed, proof tested, and calibrated offers the best solution towards reducing total system costs to future users.

To bring HTHP sampling into more common usage, further cost reductions must be achieved. Although process developers may decide to develop sampler technology and carry the burden of the system development costs government and private research agencies (DOE, EPA, EPRI, IEA, etc.) also have specific needs for such equipment. These organizations will probably carry out the technological development of these systems to the point where most of the costs have been absorbed prior to sale to the user.

Finally, there appears little doubt that emerging coal conversion technologies (PFBC, coal gasification and liquification, MHD, etc.) will require on-line HTHP extractive particulate sampling within the next 1 to 2 years. Development of advanced HTHP sampling systems need to get underway now so that they will be available at the time they are needed. The corresponding development of particulate cleanup systems are the pacing item with regard to when these sampling systems will be needed. The completion of this program is only the beginning of the development path outlined above.

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APPENDIX A

HIGH-TEMPERATURE, HIGH-PRESSURE MAILING LIST

HTHP MAILING LIST

N. Abuaf
Brookhaven National Laboratory
Upton Long Island
New York, 11973

Richard L. Adams
Wheelabrator-Frye Inc.
600 Grant Street
Pittsburgh, PA 15219

Jeffery C. Alexander
M.I.T.
Room 36-323
Cambridge, MA 02139

Gerald L. Anderson
Institute of Gas Technology
3424 South State Street
Chicago, IL 60616

Herman B. Anderson, Jr.
General Services Administration
Region 3, PBS
Technical Field Office
7th & D Streets, S.W.
Washington, D.C. 20407

Dr. John W. Anderson
Rexnord, Inc.
1914 Albert Street
Racine, WI 53404

Larry W. Anderson
Acurex/Aerotherm
485 Clyde Avenue
Mountain View, CA 94042

Stig Andersson
STAL Laval
S-61320 Finspong
Sweden

Francis M. Alpiser
Chemical Environmental Engineering
EPA III -- AHMD-SIP
Sixth and Walnut
Curtis Building MS-3A-H11
Philadelphia, PA 19106

Jim Abbott
EPA
IERL-RTP
Research Triangle Park, NC 27711

D. H. Archer
Westinghouse
Beulah Road
Pittsburgh, PA 15235

Ted Atwood
Process Engineering
Department of Energy
Room 504
20 Massachusetts Avenue
Washington, D.C. 20545

- B -

W. D. Bachalo
Spectron Development Labs.
3303 Harbor Blvd.
Costa Mesa, CA 92626

Roland Beck
Department of Energy
7374 S. Forest
Whittier, CA 90602

Robert W. Bee
Consultant
126 Hopeland Lane
Sterling, VA 21170

Albert J. Bevolc
Associate Physicist
US Department of Energy
Ames Laboratory
A205 Physics
Ames, IA 50011

Dr. Suresh P. Babu
Institute Gas Technology
3424 S. State Street
Chicago, Illinois 60616

E. T. Barrow
Ministry of the Env.
Air Resources Branch
44880 Bay Street
Toronto, Ontario MS5 128
Canada

EPA-287 (Cin.)
(4-76)

Walter A. Baxter
Environmental Elements Corp.
P. O. Box 1318
Baltimore, MD 21230

K. Bekofske
General Electric Co.
CR&D -
P. O. Box 8
Schnectady, NY 12301

Michael Beltran
Beltran Assoc., Inc.
1133 E. 35th Street
Brooklyn, NY 11210

Paul A. Berman
Westinghouse Electric Corp.
P. O. Box 9175, Lester Branch
Philadelphia, PA 19113

Dr. Samuel Bernstein
Flow Research Co.
P. O. Box 5040
Kent, WA 98031

Prem Sagar Bhardwaja, PhD
Lawrence Berkeley Laboratory
University of California
Berkeley, CA 94720

Charles E. Billings
Environmental Engineering Science
740 Boylston Street
Chestnut, MA 02167

Samuel J. Biondo
Federal Power Commission
825 N. Capitol Street, N.E., Room 5008
Washington, D.C. 20426

David Blake
Acurex/Aerotherm
485 Clyde Avenue
Mountain View, CA 94042

Dale Blann
Acurex/Aerotherm
3301 Woman's Club Drive
Raleigh, NC 27611

Frederic L. Blum
Research-Cottrell Inc.
P. O. Box 750
Bound Brook, NJ 08805

B. H. Bochenek
U. S. EPA
401 M. Street, S.W. (A-134)
Washington, D.C. 20460

Dr. R. Boericke
General Electric Co.
Building 2, Room 712
One River Road
Schnectady, NY 12345

Dr. R. H. Boll
Babcock and Wilcox
Research Center
P. O. Box 835
Alliance, OH 44601

Mr. William L. Brangers
U. S. Army Environmental
Hygiene Agency
Abeerdeen Proving Grounds, MD 21010

Ed Brooks
TRW
One Space Park Drive
Redondo Beach, CA 90278

Robert F. Brown
Research-Cottrell
P. O. Box 750
Bound Brook, NJ 08805

Warren L. Buck
Argonne National Lab.
9700 S. Cass Avenue., Building 308
Argonne, IL 60439

Charles L. Burton
Combustion Engineering
1000 Prospect Hill Road
Windsor, CT 06095

John R. Bush
Research-Cottrell
P. O. Box 750
Bound Brook, NJ 08805

Chris Busch
President
Spectron Dev. Lab.
3303 Harbor Boulevard
Costa Mesa, CA 92606

Jeffery Bradley
Research Associate
University of Wisconsin
at Milwaukee
3200 N. Cramer Street
Milwaukee, WI 53201

Beth A. Brown
Aspen Systems Corporation
20010 Century Blvd.
Germantown, MD 20767

Bruce Boggs
Amax
5020 S. Atlanta Rd. S.E.
Smyra, GA 30080

Walt Badalski
Argonne National Lab.
9700 Cass Avenue
Argonne, IL 60439

Joan Biodi
PERC
4800 Forbes Avenue
Pittsburgh, PA 15213

Eugene Baker
Univ. of N. Dakota
Grand Forks, ND 58201

- C -

John J. Casey
Sr. Marketing Engineering
Fluidyne Engineering Corp.
5900 Olson Memorial Highway
Minneapolis, MN 55422

Gilbert K. C. Chen
Monsanto
800 North Lindbergh Blvd.
St.Louis, MO 63166

John F. Cobianchi
J. P. Stevens & Company, Inc.
1185 Avenue of the Americas
New York, NY 10036

Dr. Hugh Coleman
Sandia Laboratories
P. O. Box 5800
Albuquerque, NM 87115

John Colton
TRW
7600 Colshire Drive
McLean, VA 22101

James Colyar
Process Engineering
Cogas Development Company
P. O. Box 8
Princeton, NJ 08540

Peter Costanza
Staff Scientist
Textile Research Inst.
P. O. Box 65
Princeton, NJ 08540

Steven R., Cross
Buell-Envirotech
253 N. Fourth Street
Lebanon, PA 17042

Robert A. Chronowski
MITRE Corporation
Westgate Research Park
McLean, VA 22101

Robert Carlson
Acurex/Aerotherm
485 Clyde Avenue
Mountain View, CA 94042

Captain Jesse B. Cabellon
U. S. Army Environmental
Hygiene Agency
Abeerdeen Proving Grounds, MD 21010

Dr. Seymour Calvert
Air Pollution Technology, Inc.
4901 Morena Blvd, Suite 402
San Diego, CA 92117

Dr. R. L. Carpenter
Lovelace Biomedical and Environmental
Research Institute
P. O. Box 5890
Albuquerque, NM 87115

Captain James W. Carroll
U. S. Army Environmental
Hygiene Agency
Abeerdeen Proving Grounds, MD 21010

Gregory H. S. Cheng
Environeering, Inc.
4233 N. United Parkway
Schiller Park, IL 60176

C.K.H. Choi
TRW Energy Systems Group
Building R4/Room 2136
One Space Park Dr.
Redondo Beach, CA 90278

Dr. David F. Ciliberti
Westinghouse R&D Center
Beulah Road, Bldg. 501-3E29
Pittsburgh, PA 15235

Neil H. Coates
Mitre Corp./Metrek Div.
1820 Dolly Madison Blvd.
McLean, VA 22101

Leland Collins
Mech. Eng. Dept.
Stanford University
Stanford, CA 94305

Steven D. Colome
Harvard School of Public Health
665 Huntington Avenue
Boston, MA 02115

Glenn Coury
Coury & Associates, Inc.
7400 W. 14th Avenue
Lakewood, CO 80214

Rufus Crawford
Stearns-Roger Eng. Co.
P. O. Box 5888
Denver, CO 80217

T. E. Ctrvtnicek
Monsanto-Dayton Labs
1515 Nicholas Road
Dayton, OH 45407

John S. Cvicker
Foster-Wheeler Energy Corp.
110 S. Orange Avenue
Livingston, NJ 07039

Fred Cox
Menardi Southern
1201 W. Francisco Blvd.
Torrance, CA 90502

Irving Carls
Argonne National Lab.
9700 Cass Avenue
Argonne, IL 60439

Wm. Corder
Battelle-Columbus
505 King Avenue
Columbus, OH 43201

Dr. Stanley Canada
G.E.
P.O. Box 8
Schnectady, NY 12301

- D -

John C. Dempsey
Program Manager
U.S. Department of Energy
WPR Division
Washington, D.C. 20545

Mark N. Director
Head, Gas Physics Section
Atlantic Research Corp.
5390 Cherokee Avenue
Alexandria, VA 22314

Thomas Donnelly
Donaldson Company, Inc.
P. O. Box 1299
Minneapolis, MN 55440

Frederick J. Dudek
UOP, Inc. Air Correction Div.
P. O. Box 1107, Tokeneke Road
Darien, CT 06820

John M. Ducan
Shell Development
P. O. Box 2463 -- One Shell Plaza
Houston, Texas 77001

Alberta M. Dawson
Sala Magnetics, Inc.
247 Third Street
Cambridge, MA 02142

- E -

Sally Edler
Bendix Environmental & Process
Instruments Division
1400 Taylor Avenue
Baltimore, MD 21204

Heinz L. Engelbrecht
Wheelabrator-Frye Inc.
600 Grant Street
Pittsburgh, PA 15219
Joseph Epstein
MHD Division
U.S. Department of Energy
20 Massachusetts Avenue, N.W.
Washington, D.C. 20545

George Erskine
Mitre Corp./ Metrek Division
1820 Dolly Madison Blvd.
McLean, VA 22101

Robert Ellman
Department of Energy
Grand Forks Energy Res. Ctr.
Grand Forks, ND 58202

- F -

H. N. Frock
Leeds & Northrup
Dickerson Road
North Wales, PA 19454

B. P. Faulkner
Allis-Chalmers Corp.
P. O. Box 512
Milwaukee, WI 53201

William Fedarko
U.S. Department of Energy
20 Massachusetts Avenue, NW
Washington, D.C. 20545

Paul L. Feldman
Research-Cottrell
P. O. Box 750
Bound Brook, NJ 08805

Lewis K. Felleisen
EPA, Region III
Curtis Building
Sixth and Walnut Streets
Philadelphia, PA 19106

Paul E. Fredette
Midland-Ross Technical Center
P. O. Box 907
Toledo, OH 43691

Max Friedman
Chemico Air Pollution Control Co.
One Penn Plaza
New York, NY 11801

David Ferrell
CE Power Systems
1000 Prospect Hill Rd
Windsor, CT 06095

Dale Furlong
Buell Emission Control Division
Envirotech Corp.
200 N. Seventh Street
Lebanon, PA 17042

James Facet
Facet Enterprises
434 W. 12 Mile Road
Madison Hts., MI 48071

Robert Frumerman
Frumerman Associates
5423 Darlington Road
Pittsburgh, PA 15217

Oliver Foo
Mitre/Metrek Division
Westgate Research Park
McLean, VA 22101

- G -

John A. Garbak
Montgomery County
Dept. of Env. Protection
6110 Executive Blvd., Room 340
Rockville, MD 20852

Peter Gelfand
Buell Emission Control Div.
Envirotech Corp.
200 N. Seventh Street
Lebanon, PA 17042

James A. Gieseke
Battelle-Columbus Labs.
505 King Avenue
Columbus, OH 43201

W. Giles
General Electric Co.
Corporate R&D
P. O. Box 8
Schenectady, NY 12301

Dr. Simon L. Goren
National Science Foundation
Engineering Division
Washington, D.C. 20550

John S. Gordon
TRW
Energy Systems Planning Div.
7600 Colshire Drive
McLean, VA 22101

Eugene Grassel
Donaldson Company
P. O. Box 1299
Minneapolis, MN 55440

Michael W. Gregory
Exxon Res. & Dev.
1600 Linden Avenue
Linden, NJ 07036

Ulrich Grimm
MERC
U.S. Department of Energy
P. O. Box 880
Morgantown, WV 26537

Robert Gugger
Curtiss-Wright Corporation
One Passaic Street
Wood Ridge, NJ 07075

Nigel Guilford
Ontario Res. Foundation
Sheridan Park
Mississauga, Ontario L5K 1B3

Michael Gurevich
U.S. Department of Energy
1900 S. Eads Street
Arlington, VA 22202

Karl-Axel Gustavsson
Bahco Ventilation
Bahco Systems, Inc.
P. O. Box 48116
Atlanta, GA 30362

Adi R. Guzdar
Foster-Miller Association
135 Second Avenue
Waltham, MA 02176

John A. Garbak
Environmental Engineering
Montgomery County
1909 Hyannis Ct. No. 202
McLean, VA 22101

John Gefkin
Department of Energy
Fossil Energy/Advanced Power Systems
20 Massachusetts Avenue, N.W.
Washington, D.C. 20545

Gerald M. Goblirsch
Mechanical Engineering
Department of Energy
Grand Forks Energy Research Center
Box 8213 University Station
Grand Forks, ND 58202

TYPING GUIDE SHEET

Masood Ghassemi
TRW, Bldg. R4., Rm. 1128
One Space Park Dr.
Redondo Beach, CA 90278

Stanely S. Grossel
Process Design Section
Hoffman-LaRoché, Inc.
Nutley, NJ 07110

Edw. Garruto
Curtiss-Wright Corp.
One Passaic Avenue
Wood Ridge, NJ 07075

Dr. Wm. Goldberger
Battelle-Columbus
505 King Avenue
Columbus, OH 43201

Michael Gluckman
EPRI
3412 Hillview Avenue
Palo Alto, CA 94306

- H -

H. J. Hall
H. J. Hall Associates
Cherry Valley Road
Princeton, NJ 08540

Mark S. Hanson
Battelle-Northwest
P. O. Box 999
Richland, WA 99352

Michael J. Hargrove
C-E Power Systems
1000 Prospect Hill Road
Windsor, CT 06095

G. O. Haroldsen
Allied Chemical Corporation
550 Second Street
Idaho Falls, ID 83401

Andrew Harvey
Foster-Miller Associates
135 Second Avenue
Waltham, MA 02154

William J. Havener
Waltz Mill Site Box 158
Westinghouse
Madison, PA 15663

J. H. Hedberg
Aerojet Energy Conversion Company
P. O. Box 13222
Sacramento, CA 95813

K. H. Hemsath
Surface Division
Midland-Ross Corporation
P. O. Box 907
Toledo, OH 43691

Howard E. Hesketh
Southern Illinois University
School of Eng.
Carbondale, IL 62901

R. C. Hoke
Exxon Research and Engineering
P. O. Box 8
Linden, NJ 07036

D. G. Ham
Battelle-Northwest
Battelle Blvd.
Richland, WA 99352

John D. Holmgren
Westinghouse
Waltz Mill Site, Box 158
Madison, PA 15663

Dr. Donald J. Holve
Stanford University
Mechanical Engineering
Stanford, CA 94305

Dr. Chao-Ming Huang
TVA Energy Research
1320 Commerce Union Bank Bldg.
Chattanooga, TN 37401

Gordon Huddleston
Montana Energy and MHD
Research and Dev. Inst., Inc.
P. O. Box 3809
Butte, MT 59701

EPA-287 (Cin.)
(4-76)

Jacques Hull
Acurex/Aerotherm
485 Clyde Avenue
Mountain View, CA 94042

Don Holve
Stanford University
Mechanical Engineering
Stanford, CA 94305

C. Frederick Hansen
Ames Research Center
NASA, Div. 229-3
Moffet Field, Ca 94033

Robert Hosemann
PG&E
77 Beale Street
San Francisco, CA 94106

Francine Hakimian
Librarian
McIlvaine Co.
2970 Maria Avenue
Northbrook, IL 60062

Daniel Hartley
Sandia Laboratories
Livermore, CA 94550

Donald Hull
Phillips Petroleum
P.O. Box 25
Homer City, PA 15748

Thomas Holen
Stearns-Roger
Conoco Coal Dev. Co.
Rapid City, SD 57701

Herbert I. Hollander
Gilbert Associates
P.O. Box 1498
Reading, PA 19603

- I -

C. E. Irrion
Curtiss-Wright Corp.
One Passaic Avenue
Wood Ridge, NJ 07075

- J -

Albert A. Jonke
Argonne National Lab.
9700 S. Cass Avenue
Argonne, IL 60439

I. L. Jashnani
Arthur D. Little, Inc.
One Acorn Park
Cambridge, MA 02140

Dr. Fred Jones
American Natural Service Co.
One Woodward Avenue
Detroit, MI 48226

- K -

Yale G. Kardish
Peabody Air Resources Equip. Co.
P. O. Box 5202
Princeton, NJ 08540

William M. Kelly
Environmental Elements Corp.
P. O. Box 1318
Baltimore, MD 21203

Richard A. Kennedy
Mitre/Metrek Division
1820 Dolly Madison Bovd.
McLean, VA 22101

Frank J. Kiernan
Aerojet Energy Conversion Co.
99 Clinch Avenue
Garden City, NY 11530

Richard N. Kniseley
Department of Energy
Ames Laboratory, DOE
Iowa State University
Ames, IA 50011

Dr. Charles E. Knox
Uniglass Industries
1440 Broadway
New York, NY 10018

Arthur L. Kohl
Rockwell International/
Atomics International Division
8900 DeSoto Avenue
Canoga Park, CA 91304

John J. Kovach
MERC -
Department of Energy
P. O. Box 880
Morgantown, W. VA 26505

William Krisko
Donaldson Co., Inc.
P. O. Box 1299
Minneapolis, MN 55440

Andres Kullendorff
Atal-Laval Turbin AB
S-61220 Finspong, Sweden

T. Kumar
Occidental Research Corporation
1855 Carrion Road
LaVerne, CA 91750

William B. Kuykendal
IERL-RTP
Environmental Protection Agency
Research Triangle Park, NC 27711

Henry F. Keller
Carrier Corporation/
Research Division
Carrier Parkway
Syracuse, NY 13201

Mike Klett
Process Engineering
Gilbert Associates
P. O. Box 1498
Reading, PA 19603

Harry Krokta
The Ducon Company, Inc.
147 E. Second Street
Mineola, NY 11501

Hank Kolnsberg
TRC, Inc.
125 Silas Deane Hwy.
Wethersfield, CT 06109

Dale Keairns
Westinghouse
Waltz Mill Site Box 158
Madison, PA 15663

T. Kalina
G.E.
P.O. Box 8
Schnectady, NY 12301

- L -

Norman R. LaMarche
General Electric
1 River Road
Building 23, Room 355
Schenectady, NY 12345

George Lamb
Textile Research Institute
P. O. Box 625
Princeton, NJ 08540

William T. Langan
Buell Emission Control Division
Envirotech Corporation
200 N. Seventh Street
Lebanon, PA 17042

Robert Langley
Inex Resources, Inc.
7475 W. Fifth Avenue
Lakewood, CO 80226

C. E. Lapple
Chemical Engineering Department
SRI International
333 Ravenswood Avenue
Menlo Park, CA 94025

Benjamin Linsky
A Different Air -- Skyline
1360 Anderson
Morgantown, WV 26505

C. E. Lombardi
Teller Environmental Systems, Inc.
10 Faraday Street
Worcester, MA 01605

E. T. Losin
Allis-Chalmers Corporation
Milwaukee, WI 53201

F. E. Lukens
Western Arcadia
4115 W. Ogden Avenue
Chicago, IL 60623

Bruno Loran
Ralph M. Parsons co.
100 W. Walnut Street
Pasadena, CA 91124

Vance Leak
U.S. Dept. Interior
P.O. Box 1660
Twin Cities, MN 55111

Dr. Philip Levins
Arthur D. Little, Inc.
One Acorn Park
Cambridge, MA 02140

- M -

Andrej Macek
Department of Energy
Fossil Energy/Advanced Power Systems
20 Massachusetts Avenue, N.W.
Washington, D.C. 20545

Dr. Ray F. Maddalone
TRW Defense and Space Systems Group
Building 01/2020
One Space Park Drive
Redondo Beach, CA 90278

Dr. Lidia Manson
TRW
Bldg. 01-2161
One Space Park Drive
Redondo Beach, CA 90278

William Masters
Acurex/Aerotherm
485 Clyde Avenue
Mountain View, CA 94042

I. Matsunga
Mitsubishi Heavy Industries
875 North Michigan Ave., Ste. 2100
Chicago, IL 60611

Sigvard Mats
Flakt Inc.
1500 E. Putnam Avenue
Old Greenwich, CT 06870

Mike May
Babcock and Wilcox Company
20 S. Van Buren
Barberton, OH 44203

J. T. McCabe
Mechanical Technology
968 Albany-Shaker Road
Latham, NY 12110

J. D. McCain
Southern Research Institute
2000 9th Avenue
Birmingham, AL 35205

Joseph E. McGreal
United States Steel Research
"B" Street
Penn Hills, PA 15235

James R. Melcher
MIT
Room 36-313, 50 Vassar Street
Boston, MA 02139

Dr. Arthur G. Metcalfe
Solar Turbines International
Mail Zone R-1, Box 80966
San Diego, CA 92138

Richard Mitchell
Inex Resources, Inc.
7475 W. Fifth Avenue
Lakewood, CO 80226

Mark H. Morelli
Amax Coal Company
105 S. Meridian Street
Indianapolis, IN 46225

John M. Morris
Air Pollution Division
Rexnord Inc.
P. O. Box 13226
Louisville, KY 40213

Andy Murphy
Acurex/Aerotherm
3301 Woman's Club Drive
Raleigh, NC 27611

Keshava S. Murthy
Battelle-Columbus Labs.
505 King Avenue
Columbus, OH 43201

Ron Miller
PSM Sales -- Rexnord
7675 Maple Avenue
Pennsauken, NJ 08109

William McCarthy
Chemical Engineering
US/EPA -- OEMI
Waterside Mall
Washington, D.C. 20460

George B. Manning
Department of Energy
20 Massachusetts Avenue
Washington, D.C. 20545

Dr. Jim Meyer
Research Engineering
Oak Ridge National Lab.
P. O. Box "X"
Oak Ridge, TN 37830

M. Miller
Fluidyne Engineering Corporation
5240 Port Royal Road
Springfield, VA 22151

John C. Montagna
Argonne National Lab.
9700 S. Cass Avenue
Argonne, IL 60439

R. H. Moore
Battelle-Northwest
Battelle Boulevard
Richland, WA 99352

Samuel J. Moore
Carrier Corp/Research Div.
Carrier Parkway
Syracuse, NY 13201

William E. Moore
Fossil Energy/Adv. Power Systems
Department of Energy
20 Massachusetts Avenue
Washington, D.C. 20545

Tom Mosure
US/EPA
Environmental Research Inf. Center
26 W. St. Clair
Cincinnati, OH 45268

Larry Michalec
Code 64270 NARF
NAS North Island
San Diego, CA 92135

Henry Modetz
EPA, Region IV
230 S. Dearborn
Chicago, IL 60604

M. MacCafferty
IEA Coal Research
Technical Information Service
14/15 Lower Grosvenor Place
London SWIW OEX
England

Capt. Joseph A. Martone
USAF, BSC
DET 1, HC ADTC
Tyndall AFB, FL 32403

John Miles
Phillips Petroleum
P.O. Box 25
Homer City, PA 15748

Rich McMillan
Foster-Wheeler Energy Research Ctr.
12 Peach Tree Hill Road
Livigston, NJ 07039

- N -

Leonard M. Naphtali
Department of Energy
Fossil Energy/CCN
20 Massachusetts Avenue
Washington, D.C. 20545

Charles K. Neulander
General Electric Company
P. O. Box 8, Building K-1
Schnectady, NY 12301

G. S. Newton
Lovelace Biomedical
Environmental Research Institute
P. O. Box 5890
Albuquerque, NM 87115

James C. Napier
Solar Turbines International
P. O. Box 80966
2200 Pacific Highway
San Diego, CA 92138

David V. Nakles
Carnegie-Mellon Univ.
Schenley Park
Pittsburgh, PA 15213

- O -

Thomas E. O'Hare
Brookhaven National Laboratory
Upton, Long Island, NW 11973

Dr. Morris S. Ojalvo
National Science Foundation
Engineering Division
Washington, D.C. 20550

John M. Ondov
Lawrence Livermore Lab.
P. O. Box 808
Livermore, CA 94550

Dr. H. H. Osborn
C-E Air Preheater Co.
P. O. Box 372
Wellsville, NY 14895

Dr. Nancy O'Fallon
Argonne Natl. Lab.
Applied Physics Dept., Bldg. 316
9700 Cass Avenue
Argonne, IL 60439

- P -

Subhash S. Patel
Hittman Associates, Inc.
9151 Rumsey Road Building
Columbia, MD 21045

Dr. Ronald G. Patterson
Air Pollution Technology, Inc.
4901 Morena Boulevard, Suite 402
San Diego, CA 92117

Dr. Richard D. Parker
Air Pollution Technology, Inc.
4901 Morena Blvd., Suite 402
San Diego, CA 92117

Walter Podolski
Argonne National Lab.
9700 S. Cass Avenue
Argonne, Illinois 60439

James B. Padden
Facet Filters
434 W. 12 Mile Road
Madison Heights, MI 48071

Joseph R. Polek
Catalytic, Inc.
1500 Market Street, CSW-10
Philadelphia, PA 19102

Duane H. Pondius
Southern Research Inst.
2000 9th Avenue S.
Birmingham, AL 35205

Wm Penson
Battelle-Columbus
505 King Avenue
Columbus, OH 43201

Jerry Peterson
G.E.
P.O. Box 8
Schnectady, NY 12301

Lyle Pollack
Phillips Petroleum
154 RB 1
Bartlesville, OK 74004

John Piatt
Pittsburgh and Midway Coal Company
(PAMCO)
P.O. Box 199
DuPont, WA 98327

Peter Palmer
G.E.
P.O. Box 8
Schnectady, NY 12301

- Q -

Sandra Quinlivan
TRW
Bldg. R4, Rm. 1120
One Space Park Drive
Redondo Beach, CA 90278

- R -

Ronald Renko
Development Engineering
C-E Air Preheater
Wellsville, NY 14895

D. L. Reid
Battelle-Northwest
P. O. Box 999
Richland, WA 99352

George Rey
Office of R & D (RD-681)
Env. Protection Agency
Washington, D.C. 20460

George Rinard
Research Engineer
Denver Research Inst.
Denver, CO 80208

Stephen N. Rudnick
Harvard School of Public Health
665 Huntington Avenue
Boston, MA 02115

E. Radhakrishnan
Battelle-Columbus Labs.
505 King Avenue
Columbus, Ohio 43201

Madhav B. Ranade
Research Triangle Institute
P. O. Box 12194
Research Triangle Park, NC 27709

David L. Raring
Sonic Development Corporation
3 Industrial Avenue
Upper Saddle River, NJ 07458

Dr. Richard Razgaitis
Ohio State University
206 W. 18th Avenue
Columbus, Ohio 43221

R. B. Reif
Battelle-Columbus Labs.
505 King Avenue
Columbus, Ohio 43201

Frank G. Rinker
Midland-Ross
P. O. Box 907
Toledo, Ohio 43691

Thomas J. Robertazzi
Facet Enterprises
6521 Arlington Boulevard
Falls Church, VA 22042

David R. Rubin
U.S.D.A., Rural Electrification Admin.
Agriculture South Building
14th and Independence Avenue, S.W.
Washington, D.C. 20250

Mr. Jean V. Remillieux
Air Industrie
19, Avenue DuBonnet
92401 Courbevoie
France

George L. Roberts
Vice President
Universal Transport Systems, Inc.
2665 Marine Way
Mountain View, CA 94040

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James Ronning
Curtiss-Wright Corp.
One Passaic Avenue
Wood Ridge, NJ 07075

- S -

William A. Standstrom
Institute of Gas Technology
3424 South State Street
Chicago, IL 60616

Gene Schaltenbrand
C-E Preheater Company
P. O. Box 387
Wellsville, NY 14895

Robert Scheck
Stearns-Roger
P. O. Box 5888
Denver, CO 80217

Martin Schiller
CSI Engineering
P. O. Box 1515
Fairfield, CT 06430

J. W. Schindeler
John Zink Company
P. O. Box 7388
Tulsa, OK 74105

Dr. Schnittgrunt
Consolidated Aluminum
2302 Weldon Way
St. Louis, MO 63178

H. F. Schulte
Rexnord, Inc.
1914 Albert Street
Racine, WI 53404

Gernot Mayer Schwinning
Lurgi Apparate-Technik GmbH
Postfash 1, Gwinner Street
6000 Frankfort am Main-2
West Germany

Dr. David S. Scott
Dept. of Mech Engineering
University of Toronto
Toronto, Ontario M5S 1A4
Canada

Stanley J. Selle
Grand Forks Energy Res. Ctr.
Department of Energy
P. O. Box 8213, University Station
Grand Forks, ND 58202

Michael Shackleton
Acurex/Aerotherm
485 Clyde Avenue
Mountain View, CA 94042

J. K. Shah
Surface Division
Midland-Ross Corporation
P. O. Box 907
Toledo, OH 43691

Jer-Yu Shang, Ph.D
Mitre Corp./Metrek Division
Westgate Research Park
McLean, VA 22101

W. J. Sheeran
General Electric Company
Corporate R&D
P. O. Box 43
Schenectady, NY 12301

Dr. Thomas S. Shevlin
3M Company
3M Center, P. O. Box 33221, Bldg. 230
St. Paul, MN 55138

Dr. Gajendra H. Shroff
Bechtel Power Corporation
15740 Shady Grove Road
Gaithersburg, MD 20760

Dr. A. P. Sikri
U.S. Department of Energy
Gas & Shale Technology
20 Massachusetts Avenue
Washington, D.C. 20545

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A. A. Smith
Babcock and Wilcox Ltd.
Woodall-Duckham House
Crawley, Sussex,
United Kingdom

Dr. Donald W. Smith
HAVEG Industries
900 Greenbank Road
Wilmington, DE 19808

Wallace B. Smith
Southern Research Inst.
2000 9th Avenue
Birmingham, AL 35205

John H. Smithson
Department of Energy
20 Massachusetts Avenue
Washington, D.C. 20545

Herbert W. Spencer, III
Western Precipitation Division
Joy Manufacturing
4565 Colorado Boulevard
Los Angeles, CA 90039

John Spriggs
Donaldson Company, Inc
P. O. Box 1299
Minneapolis, MN 55440

Dr. C. J. Stairmand
Babcock and Wilcox Ltd.
Woodall-Duckham House
Crawley, Sussex
United Kingdom

Richard C. Stone
Stone & Webster Engineering Company
P. O. Box 2325
Boston, MA 02107

William M. Swift
Argonne National Laboratory
9700 S. Cass Avenue, Bldg. 205
Argonne, IL 60439

Ronald D. Synder
Buell Emission Control Division
Envirotech Corporation
200 N. Seventh Street
Lebanon, PA 17042

Robert K. Schaplowsky
Chemist
Aerojet
7767 LaRivera Drive No. 219
Sacramento, CA 95826

David Shaw
Professor
State University of NY/Buffalo
4232 Ridge Lea Road
Buffalo, NY 14226

G. F. Schiefelbein
Battelle-Northwest
Battelle Boulevard
Richland, WA 99352

Jer Yu Shang
Professional Engineering
4524 Andes Drive
Fairfax, VA 22030

Kevin J. Shields
Hittinaw Associates, Inc.
9190 Red Branch Road
Columbia, MD 21045

Jack Siegel
Department of Energy
Fossil Energy/Advanced Power Systems
20 Massachusetts Avenue
Washington, D.C. 20545

Theodore B. Simpson
Department of Energy
Fossil Energy/Advanced Power Systems
20 Massachusetts Avenue
Washington, D.C. 20545

Gregory W. Smith
Argonne National Lab.
9700 S. Cass Avenue
Argonne, IL 60439

Walter Steen
Chemical Engineering
US EPA
IERL-RTP
Research Triangle Park, NC 27711

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Alex Turchine
Proctor and Gamble
7162 Reading Road
Cincinnati, OH 45222

Suresh P. Tendulkar
Westinghouse
Waltz Mill Site, Box 158
Madison, PA 15663

C. M. Thoennes
General Electric Company
Building 2, Room 712
One River Road
Schnectady, NY 12345

Dr. Jim Trolinger
Spectron Development Labs
3303 Harbor Boulevard
Costa Mesa, CA 92626

S. I. Taub
IU Conversion Systems, Inc.
Joshua Road and Stenton Avenue
P.O. Box 331
Plymouth Meeting, PA 19462

Charles Trilling
Rockwell/Atomics International
P.O. Box 319
Canoga Park, CA 91304

- U -

V. S. Underkoffler
Manager, Combustion and Advanced Power
Gilbert Associates, Inc.
525 Lancaster Avenue
Reading, PA 19603

- V -

V. A. Varady
UOP Process Division
UOP Plaza
Des Plaines, IL

E. S. Van Valkenburg
Leeds and Northrup Company
Dickerson Road
North Wales, PA 19454

F. Munro Veazie
Owens-Corning Fiberglass Corporation
Technical Center
Granville, OH 43023

Geza Vermes
Westinghouse
1500 Chester Pike, Building A703
Eddystone, PA 19013

John A. Verrant
Donaldson Company, Inc.
P. O. Box 1299
Minneapolis, MN 55440

S. N. Vines
University of VA, Chemical Eng. Dept.
Thornton Hall
Charlottesville, VA 22901

G. J. Vogel
Argonne National Lab.
9700 S. Cass Avenue
Argonne, IL 60439

D. V. Vukovic
Faculty of Technology and Metallurgy
Belgrade University
1100 Beogard, Karnegijeva 4,
P.O.B. 494
Yugoslavia

- W -

Gordon L. Wade
Combustion Power Company
1346 Willow Road
Menlo Park, CA 94025

Donald E. Wambsgans, II
District of Columbia
Dept. of Environmental Services
Room LL-3
614 "H" Street N.W.
Washington, D.C. 20001

Walter Wolowodivk
Foster-Wheeler Dev. Corp.
12 Peach Tree Hill Road
Livingston, NJ 07039

Peter Waldstew
Physical Chemist
RDR
P. O. Box 33128
District Heights, MD 20028

Andrew Wallo
Env. Scientist
1605 Craig Street
Sterling, VA 22170

Phillip C. White
Department of Energy
Fossil Energy/Advanced Power Systems
20 Massachusetts Avenue, N.W.
Washington, D.C. 20545

Stephen Wander
Department of Energy
400 First Street, N.W.
Washington, D.C. 20545

Val E. Weaver
Department of Energy
Fossil Energy/Advanced Power Systems
20 Massachusetts Avenue, N.W.
Washington, D.C. 20545

Frank Weiskopf
Environmental Elements Corp.
P. O. Box 1318
Baltimore, MD 21203

Clyde L. Witham
SRI International
333 Ravenswood
Menlo Park, CA 94025

Thomas Walsh
Riely-Stoker
P.O. Box 547
Worcester, MA 01613

David Weller
Curtiss-Wright Corp.
One Passaic Avenue
Wood Ridge, NJ 07075

D. E. Woodmansee
G.E.
P.O. Box 8
Schnectady, NY 12301

Dr. John S. Wilson
Department of Energy/MERC
P.O. Box 880
Morgantown, WV 26505

- Y -

Dr. H. C. Yeh
Lovelace Biomedical and Environmental
Research Institute
P. O. Box 5890
Albuquerque, NM 87115

- Z -

Dr. Karim Zahedi
President
EFB, Inc.
94 Francis Street
Brookline, MA 02146

John H. Zarnitz
N.Y.C. Dept. of Air Resources
7044 Manse Street
Forest Hills, NY 11375

Dr. F. A. Zenz
The Ducon Company
147 East Second Street
Mineola, LI, NY 11501

August H. Zoll
Curtiss-Wright Corporation
Power Systems
Wood Ridge, NJ 07075

Stephen H. Zukor
Department of Energy
OGST
20 Massachusetts Avenue, N.W.
Washington, D.C. 20545

Irena M. Zuk
Interdevelopment, Inc.
Suite 104,
Rutherford B. Hayes Bldg.
2361 South Jefferson Davis Hwy.
Arlington, VA 22202

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APPENDIX B

SURVEY OF CONTINUOUS PARTICULATE MEASUREMENT DEVICES

B.1 SUMMARY

This investigation surveys the state-of-the-art of continuous particulate monitoring devices applicable to high-temperature and high-pressure (HTHP) processes. The study determined that continuous particulate monitoring of HTHP-process streams, involving fluidized bed combustion and coal gasification, are relatively new frontiers for particulate sampling technology. No commercial monitoring device is currently available that can meet this need; however, research efforts are underway to develop the technology.

Instrumentation based on electro-optics is very attractive for this future need. In addition to the electro-optical devices, the IKOR continuous monitor, based on the principle of contact charge transfer, also is a potential candidate for HTHP monitoring.

B.2 INTRODUCTION

The deficiencies of existing monitoring equipment is a hindrance to process control and environmental protection in the development of advanced coal and energy conversion systems, (e.g., fluidized bed combustors and coal gasifiers).

In particular, Argonne National Laboratories has concluded (Reference B-1) that coal-fired processes (both existing and proposed) require instrumentation development for the monitoring of:

- Mixed phase flowrates of dense phase solids mass flow, dilute phase solids mass flow, and dirty gas flow
- On-line composition of streams (containing solids, liquids, and gases) and vessels (containing solids and liquids)
- Level detection of oil/water interfaces occurring in coal conversion processes and of fluidized-bed heights
- Rapidly fluctuating temperatures, in the 1500°F to 3000°F range

Continuous particulate sampling is also essential to the determination of on-line process conditions. The importance of this sampling is underscored for HTHP streams, which can corrode or erode exposed equipment surfaces such as turbine blades.

This investigation presents a brief survey of the state-of-the-art of continuous particulate monitoring equipment. Information sources include current literature and equipment manufacturers.

The coal energy conversion industry encompasses a variety of processes which can span a wide range of operating conditions. This survey considers the sampling environment defined below, which is representative of technology currently under development.

- Temperature: 1200^{o}F to 2000^{o}F
- Pressure: 3 to 100 atm
- Flow velocity: 10 to 100 ft/sec
- Particulate size: $<60\ \mu m$
- Particulate loading: 0.04 to 4.4 gram/m^3

Factors which create hostile environments for particulate monitoring equipment are:

- The existence of high temperature and high pressure
- Corrosive, abrasive, and highly flammable nature of the constituents, while nonuniform flow profiles complicate the determination of particulate loading.

B.3 DISCUSSION

The current state of particulate detection technology is comprised of continuous monitoring and noncontinuous sampling. Virtually all of the hardware for measurements at modest temperatures and pressures falls in the latter category. Typical techniques consist of sample extraction, sample conditioning, sample collection (filters, impactors and cyclones) and subsequent weighing of this sample in a gravimetric balance. Reference 2 presents a survey of the existing noncontinuous HTHP sampling techniques.

As mentioned previously, the requirement for continuous monitoring equipment is critical to the development and subsequent commercialization of advanced coal energy systems. This document presents an overview of the state-of-the-art of continuous particulate monitoring at HTHP, which is still in the very initial stages of development. As such, the approach here looks at the existing continuous particulate monitoring techniques and presents their status for HTHP application. The field of continuous monitoring divides into two categories: extractive and nonextractive sampling. A discussion of these is presented below.

B.3.1 Extractive Continuous Monitoring

The methods studies using the extractive monitoring approach were based upon:

- The Piezo-electric effect
- Beta wave attenuation
- Contact charge transfer (tribo-electric effect)

B.3.1.1 Piezo-Electric Effect

Instruments using this method are called piezo-electric microbalances. The heart of the microbalance is a quartz crystal, which acquires a particulate sample deposition by impaction (see Figure B-1). This alters the natural frequency of vibration of the crystal, and the shift in frequency correlates with the particulate mass. Reference 3 presents a detailed discussion of this technique.

At the particulate loadings of interest for HTHP process stream measurements, this technique is essentially a noncontinuous one. Piezo-electric microbalances monitor low concentrations at ambient conditions. Dr. Wallace of Berkeley Industries (Reference 4) advised that the current limit for these instruments is a frequency shift of 2000 Hz. This implies a mass concentration of about 150 $\mu g/m^3$ for 30 minutes without overloading the crystal. In addition, the frequency response of the crystal is very sensitive to both pressure and temperature, an undesirable trait for HTHP application. As such, this technique was deamed not suitable for HTHP process steam monitoring.

B.3.1.2 Beta-Wave Attenuation

Figure B-2 presents a Beta-wave radiation attenuation instrument and its use as an emissions mass monitor. Differences in Beta-wave transmission between the clear and loaded portions of a filter tape determine the radiation attenuation due to the presence of particulate. The particulate mass is directly related to this attenuation. A detailed discussion of this technique is presented in Reference 3.

A recent investigation conducted by C. H. Gooding (Reference 5) found the GCA instrument based on Beta-attenuation to be inaccurate. He, along with others, have stressed that further work is needed to improve conversion from Beta-attenuation to particulate mass. Besides this, the current temperature limit for filter tapes is about 600°F, and the high pressure is likely to pose a serious gas leakage problem. Hence, this method was also judged unsuitable for HTHP requirements.

B.3.1.3 Contact Charge Transfer (Tribo-Electric Effect)

If a particle can be made to hit or slide along a surface, it will usually transfer electrical charge to the surface. This creates a voltage potential which will cause a current (10^{-7} to 10^{-8} amps, typically) within conducting materials. This is one example of the tribo-electric effect (Reference 3). Figure B-3 illustrates the use of the IKOR continuous monitor, which is based on this principle.

Two companies produce(d) particulate sampling equipment based on this principle: IKOR and KONITEST* (German). IKOR markets both an extractive and a nonextractive sampler. This type of monitor holds promise for application to HTHP process stream monitoring.

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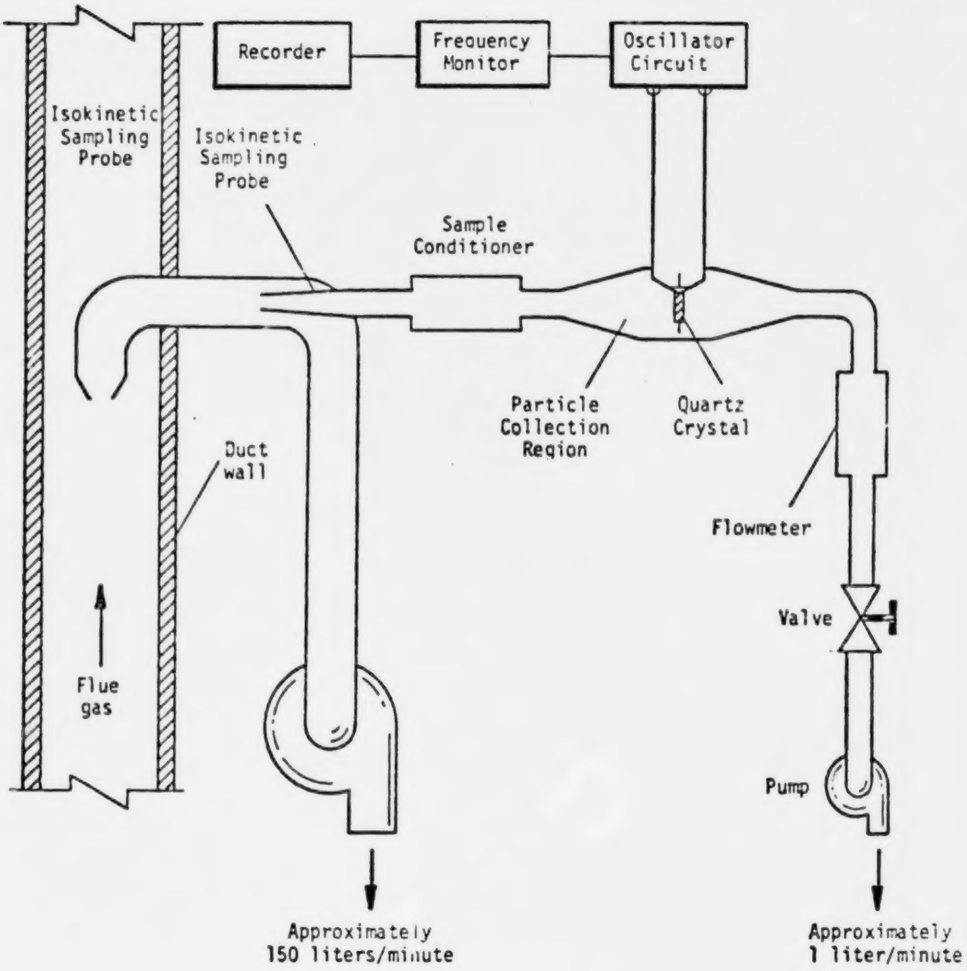
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Frequency Monitor
Oscillator Circuit
Isokinetic Sampling Probe
Isokinetic Sampling Probe
Sample Conditioner
Particle Collection Region
Quartz Crystal
Duct wall
Flowmeter
Valve
Flue gas
Pump
Approximately 150 liters/minute
Approximately 1 liter/minute

Figure B-1. Piezo-electric microbalance (quartz) particulate sampling train.

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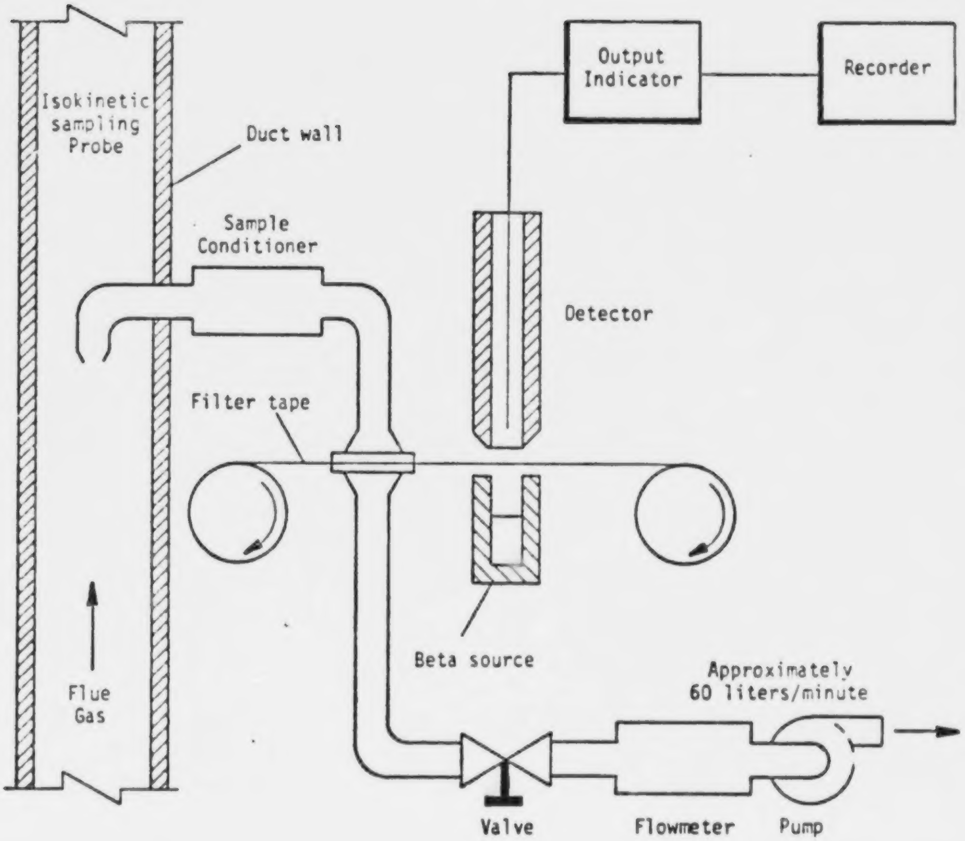
Isokinetic sampling Probe
Duct wall
Sample Conditioner
Output Indicator
Recorder
Detector
Filter tape
Beta source
Approximately 60 liters/minute
Flue Gas
Valve
Flowmeter
Pump

Figure B-2. Beta-wave attenuation particulate sampling train.

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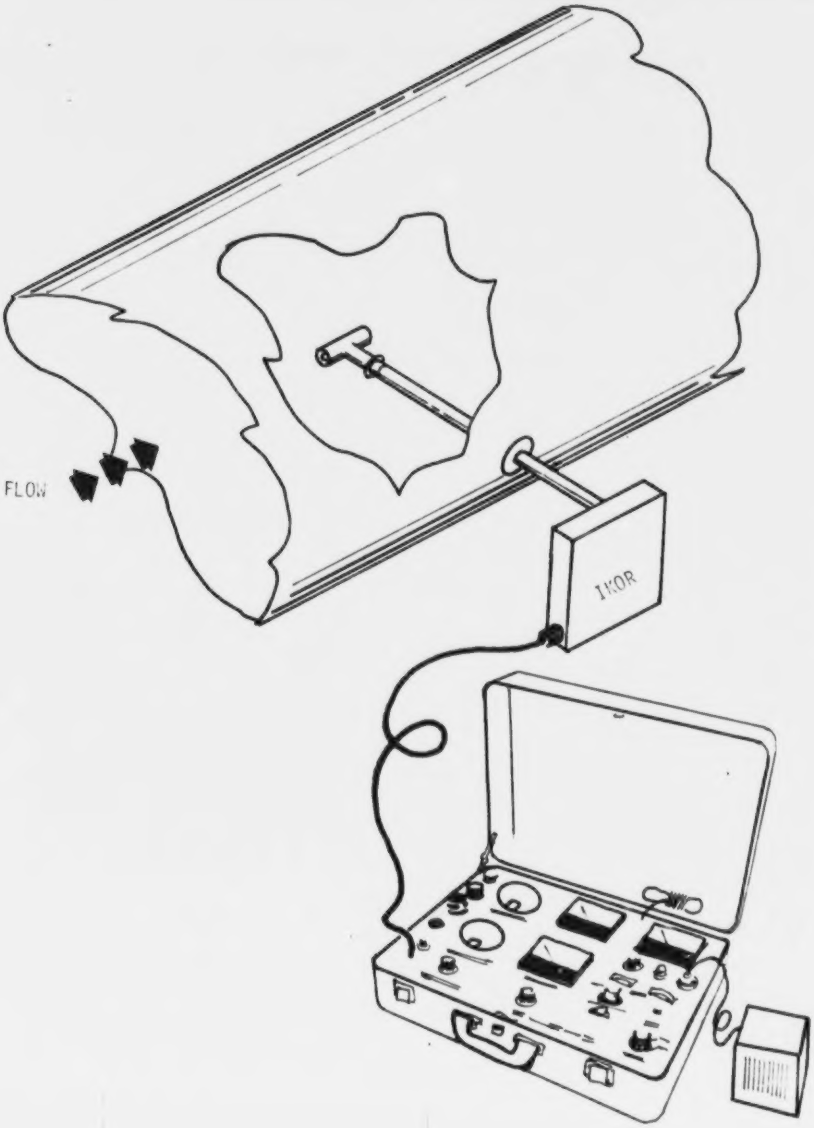

Figure B-3. IKOR continuous particulate mass monitor based on contact charge.

According to the equipment specifications, the IKOR probe can detect mass concentrations from 1 x 10^{-4} grains/scf to 100 grains/scf for particles between 0.1 and 100 µm in size. Mr Gruber of IKOR (Reference 6) stated that the IKOR samplers have been used at high temperatures (2000°F) and low pressure (ambient) and low temperatures and high pressures (350 psi). He further indicated that the corrosive environment of these process streams of interest will not damage a suitable probe material, such as Titanium.

Studies recently conducted (Reference 7) show that with the IKOR continuous monitor particulate concentration readings are a strong function of flow velocity. Further testing and evaluation are needed to resolve this problem.

B.3.2 Nonextractive Continuous Monitoring

Since HTHP process streams are corrosive, abrasive and volatile in nature, nonextractive monitoring is preferable to minimize the potential of system leaks, which could be hazardous. In this category the discussion will center on methods using electro-optics.

B.3.2.1 Electro-Optical Methods

This field of the particulate sampling technology is currently receiving much attention. A variety of instrumentation is under development to determine the size, number density, mass concentration and velocity of the particulates. These techniques, in which the particulates are illuminated by a beam of light (e.g., laser or LED), divide into the two categories listed below:

- Light absorption
- Light scattering

Instruments based on the former principle are opacity meters, or transmissometers. They rely upon correlations of opacity versus mass concentration, and are classified as "crude" techniques; hence, this investigation did not concentrate on them in detail. However, they appear to be useful for determining relative changes in particulate loading as an aid to process control.

Instrumentation based on light scattering are sophisticated diagnostics approaches that may be capable of resolving particulate size distributions as well as loading densities. These devices rely on application of the MIE Theory (References 8 and 9), which describes the scattering of electromagnetic radiation from particles. For a description of the theory and its applications, the reader is referred to recent investigations by Coleman, et al., (Reference 10).

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*Not in production since 1968.

Applications of light scattering for diagnostics has burgeoned into a vast technology which continues to expand. It is neither possible nor desirable to present a comprehensive review in this study. What is presented is a discussion of techniques which have the greatest potential application to HTHP processes. These are based upon

- Photography
- Holography
- Visibility
- Intensity Ratio
- Diffraction

Photography is the simple camera technique using a spark or pulsed laser light source (Figure B-4). References 11 and 12 provide details of specific systems. Advantages of the photographic technique are insensitivity to refractive index, applicability to heavy particle loading densities, optical simplicity, the ability to resolve size and shape for particles greater than 2 micrometers in diameter, and real time information gathering. However, the method suffers from a limited sensitivity to small particles and narrow depth-of- fields at large magnifications. Furthermore, data reduction is an extremely time-consuming process.

Figure B-5 illustrates the formation of a hologram by the splitting and subsequent coalescence of a laser beam. Light scattering causes a phase shift in the light passing through the flowfield. Hence, when this beam recombines with the reference beam, the phase difference creates an interference pattern. McCreath and Beer (Reference 13) present the analysis relating this pattern to particle size. Holography has little or no depth-of-field restriction and assures that magnification of particles along the optical axis is uniform. It can measure particle sizes $>5\,\mu$ diameter. However, this method requires complex equipment and painstaking adjustment; moreover, size information cannot be obtained in real time.

The visibility technique is an application of the fact that a particle will modulate as well as cause a Doppler-shift in the frequency of a laser beam. Figure B-6 shows a typical system used to obtain visibility information. A laser beam is split into two equal beams, which are focused and intersect in the region of interest (called a measurement volume). Interference of these beams form a fringe pattern, which produces a modulated and shifted signal when perturbed by a particle traversing this volume. The particle diameter determines the maximum and minimum of this signal (Reference 14).

Hodkinson (Reference 15) first proposed to determine particle size by using the scattered light intensities from two, or more, directions (i.e., Θ_1, Θ_2, ..., Θ_n; Figure B-7). In particular, this method (the intensity ratio technique) is an application of the relationship between the ratio of two such intensities, their Θ_i's, and the particle size. Gravatt (Reference B-16) extended this technique to cover a range of refractivities, while Hirleman, et al., (Reference 7) have developed a two-ratio modification of the technique. The intensity ratio(s) technique is able to provide real

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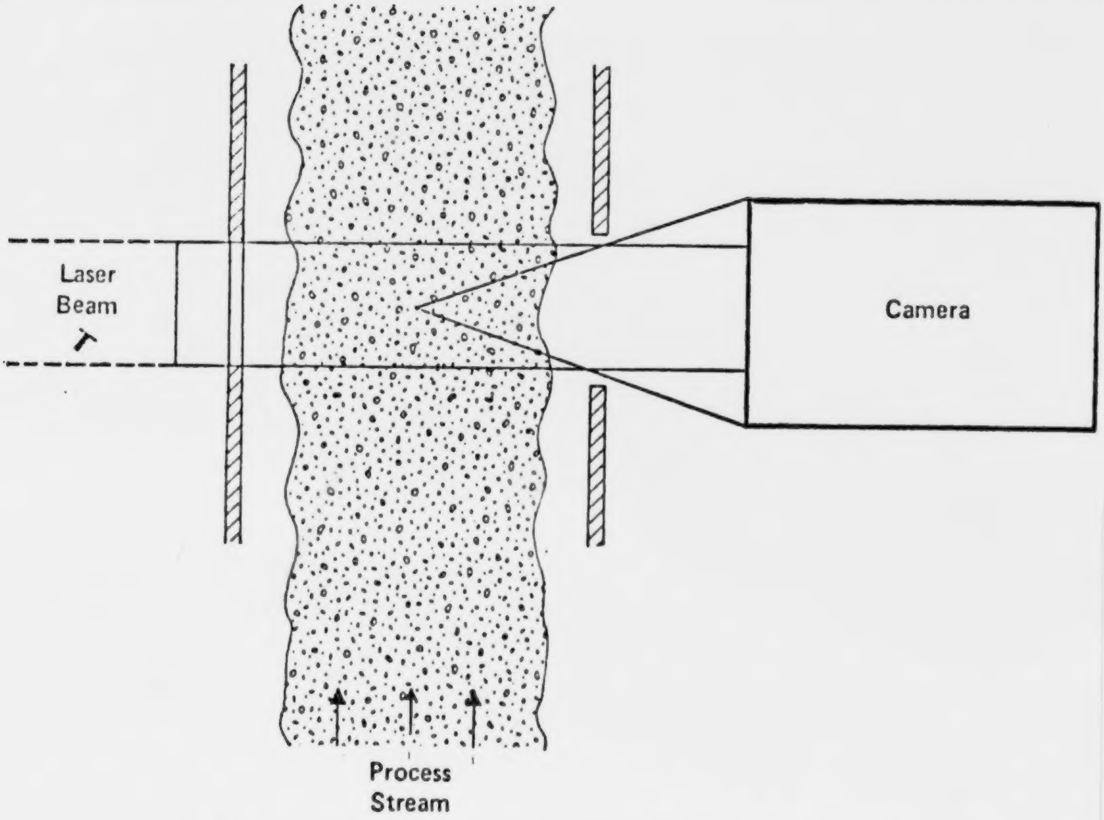
Laser Beam
Camera
Process Stream

Figure B-4. Photography technique.

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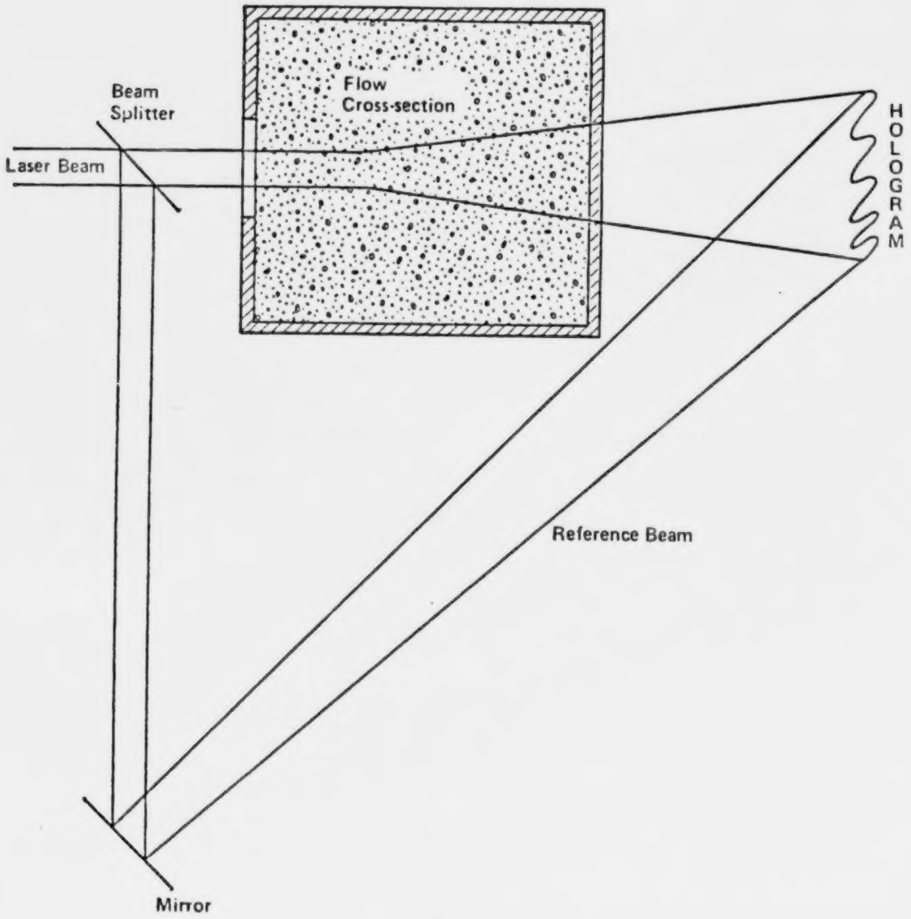

Flow
Cross-section
Beam
Splitter
Laser Beam
HOLOGRAM
Reference Beam
Mirror

Figure B-5. Holographic technique.

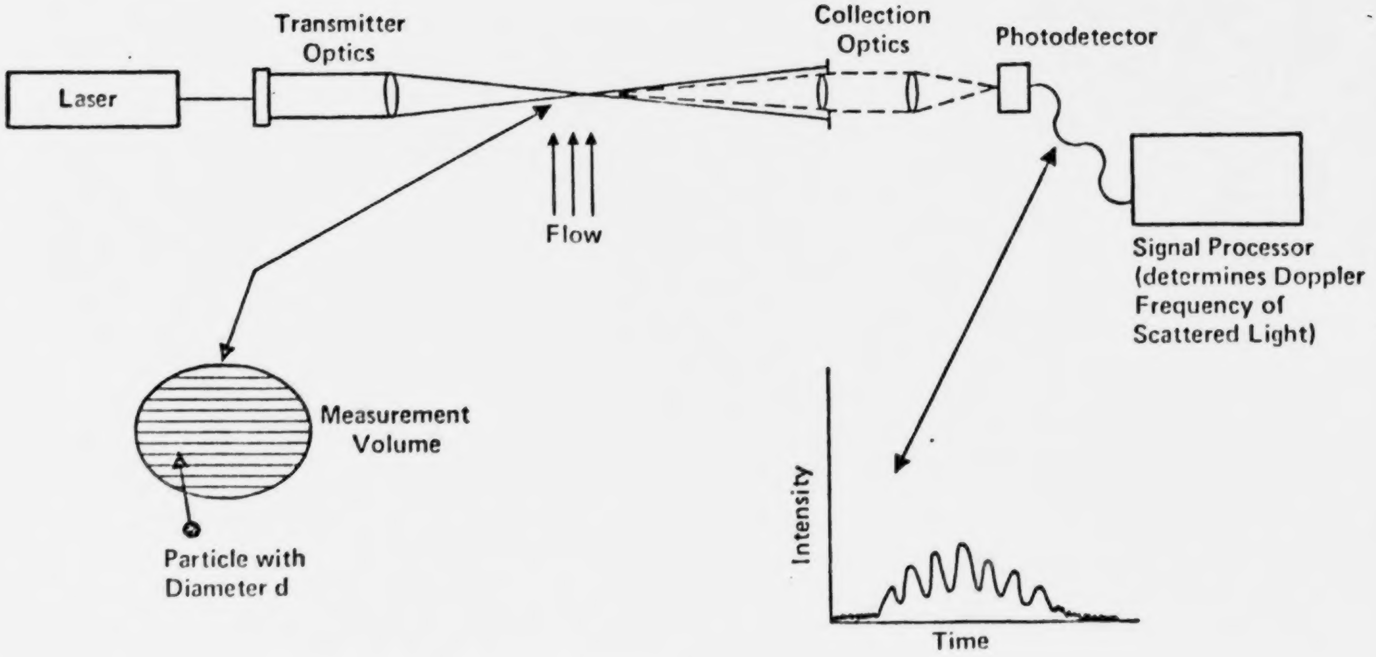
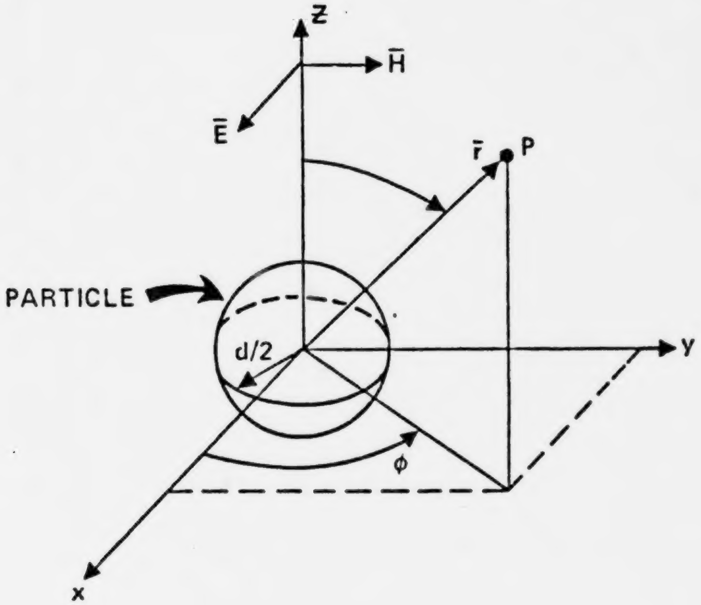

Laser
Transmitter Optics
Flow
Collection Optics
Photodetector
Signal Processor (determines Doppler Frequency of Scattered Light)
Measurement Volume
Particle with Diameter d
Intensity
Time

Figure B-6. Visibility technique.



Z – direction of propagation of linearly polarized incident light

$\overline{E}$ – direction of electric vector

$\overline{H}$ – direction of magnetic vector

$\overline{r}$ – direction of scatter to the point of observation (P)

Figure B-7. Scattering of electromagnetic radiation.

time information and apparently is not overly sensitive to either particle shape or refractivity. However, it is limited to loading densities low enough to provide single-particle signals.

The diffraction technique determines the size distribution of particles within the cylindrical volume formed by a laser beam passing through a flowfield; this method uses the total Fraunhofer diffraction pattern from the particles within the volume. McCreath and Beer (Reference 13) and Swithenbank, et al., (Reference 18) describe the technique and present experimental results. The method is optically simple, provides real time information, and is insensitive to particle shape. Its drawbacks are that it is limited to particle diameters $>2\mu$, requires large measurement volume, and is a line-of-sight rather than a point measurement technique. This method also requires a prior assumption as to the shape of the size distribution curve.

With the exception of the diffraction technique, any of the above methods can supply information to determine loading densities. Number densities are a function of size distribution and flowrate through a known volume. However, since particles may be porous or hollow, conversion of number densities to mass densities is uncertain. In fact, the only reliable method to determine mass loadings is to withdraw a sample and weigh it.

The electro-optical monitoring technology is an attractive candidate for HTHP application. However, there are some challenging hurdles to be overcome. The "window" concept is a key issue and its applicability needs to be demonstrated in terms of sealing the window area for leaks and for keeping the window clean from tar and slag deposits. One current proposal is to use either a quartz or sapphire window.*

Another method proposed is to eliminate the window altogether. Instead, the light source is housed in a sealed containment vessel outside of the flow duct. A hole is cut in the duct wall to allow for beam transmission. Hot gases are kept from entering the containment vessel by continuously pressurizing the vessel with an inert gas.

Secondly, the ability of these techniques to overcome some of the HTHP sampling environment aspects must be domonstrated, especially to background light radiation** interference from the hot gases, and the opacity of the stream itself.

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*These windows are well known to "fog-up" (crystallize) at high temperatures; however, this is not likely to be a serious problem for this application if sapphire is used. Quartz is not recommended over 1500°F.

**Initial estimates show that gas radiation is not likely to be a problem but wall radiation is a potential source of interference.

B.3.2.2 Manufacturers

There are currently four major manufacturers of this type of equipment, all of whom were contacted during this study.

1. Environmental Systems Corporation (ESC), Knoxville, Tennessee, Dr. Hal Schmitt (PILLS System).
2. Particle Measuring Systems, Inc. (PMS), Boulder Colorado. Dr. Bob Knollenberg (ASAS System).
3. Spectrom Development Labs (SDL), Tullahoma, Tennessee. Dr. Mike Farmer (MORPHOKINETOMETER System).
4. Leeds and Northrup Company (L&N), North Wales, Pennsylvania. Dr. Emil C. Muly (Microtract System).

All of these manufacturers produce equipment which have the capability to monitor particle size and mass density in the range of interest. In addition, the Spectron device has the capability of measuring particle velocity using the laser doppler system. However, at this time all of these manufacturers lack experience in applying their technology to the problem of HTHP process stream monitoring. Both Spectron Labs and Leeds & Northrop are currently under contract to DOE to develop a suitable optical system under test conditions at Argonne National Laboratory. Sandia Laboratory (Livermore, California) is also under contract to DOE to evaluate several optional devices under bench test conditions. However, work is just getting underway at the time of this study.*

B.4 CONCLUSIONS

This state-of-the-art survey indicates that continuous particulate monitoring of high temperature and pressure process streams is a new frontier for sampling technology. No commercial particulate monitoring instrument is currently available which can meet this need. However, efforts are underway to develop the technology.

This investigation indicates that the optical particulate monitoring technique appears to be very attractive for this need. Some of the key issues that need to be addressed are:

- Applicability of the "window" concept
  - -- Sealing the window area for leaks
  - -- Keeping the window clean from slag and tar deposits
  - -- Integrity of window glass (quartz or sapphire) under the HTHP environment

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*Significant testing has occurred since this section was written. The reader should contact the manufacturers for the status of their development program.

- Ability to accurately monitor in the presence of
  - -- High velocity and high levels of opacity
  - -- Radiation interference from the walls

The results of current DOE contract efforts of Spectron Labs, Leeds, & Northrup, and Sandia Laboratories should provide answers to some of these questions. In addition to the optical techniques, the IKOR continuous monitor also appears to be a potential candidate for HTHP monitoring. The key issues that need to be resolved for this device are:

- Choice of probe material
- Dependence of mass concentration readings on flow velocity

Here again some actual tests should be undertaken to address these issues. It is also recommended that light transmissometer technology be looked at in greater detail for possible application to HTHP monitoring needs.

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